## QUALITY ASSURANCE PROJECT PLAN

# UPRIVER REACH BACKGROUND INVESTIGATION LOWER WILLAMETTE RIVER

Prepared for

### **OREGON DEPARTMENT OF ENVIRONMENTAL QUALITY**

PORTLAND, OREGON January 15, 2021 Project No. 0785.13.01



Prepared by Maul Foster & Alongi, Inc. 3140 NE Broadway Street, Portland, OR 97232

#### QUALITY ASSURANCE PROJECT PLAN

UPRIVER REACH BACKGROUND INVESTIGATION LOWER WILLAMETTE RIVER

The material and data in this plan were prepared under the supervision and direction of the undersigned.

Michae	V Pickering,	RG
Maul 1	Foster & A	longi Program Manager

Date: January 15, 2021

Joshua Elliott, PE Maul Foster & Alongi Project Manager

January 15, 2021

Erik Naylor

Maul Foster & Alongi Quality Assurance Manager

Date: January 15, 2021

Carolyn Wise, RG

Maul Foster & Alongi Field Coordinator

January 15, 2021

Meaghan Pollock, RG Maul Foster & Alongi Field Coordinator

Date: January 15, 2021

Mark Pugh

Oregon Department of Environmental Quality Task Manager

Madi Novak

Madi Novak

U.S. Environmental Protection Agency Remedial Project Manager

Date: \_Jan 21, 2021

Karin Feddersen-Lethe
Karin Feddersen-Lethe (Jan 21, 2021 15:33 PST)

Donald Brown

U.S. Environmental Protection Agency Quality Assurance Manager

Date: Jan 21, 2021

Phil Wiescher, PhD

Maul Foster & Alongi Project Scientist

January 15, 2021 Date:

Mary Benzy

Maul Foster & Alongi Database Manager

Date: \_\_ January 15, 2021

## CONTENTS

TABLES	AND II	LUSTRATIONS	V
ACRONYMS AND ABBREVIATIONS			VI
1		DUCTION QUALITY ASSURANCE OBJECTIVES SCOPE AND PURPOSE	1 1 2
2	2.1	ECT AND TASK ORGANIZATION PROJECT TEAM ORGANIZATION ANALYTICAL LABORATORY RESPONSIBILITIES SAMPLING VESSEL SUBCONTRACTOR RESPONSIBILITIES	2 2 5 6
3	3.1 3.2	ECT DESCRIPTION PROJECT DATA NEEDS AND SCOPE SAMPLE PROGRAM DESIGN AND RATIONALE PARAMETERS TO BE TESTED PROJECT SCHEDULE	6 6 7 7
4	4.1 4.2 4.3 4.4 4.5 4.6 4.7 4.8 4.9 4.10 4.11 4.12 4.13	GENERATION AND ACQUISITION DATA QUALITY OBJECTIVES SAMPLE PROCESS DESIGN SAMPLING METHODS SAMPLE HANDLING LABORATORY PROCEDURES ANALYTICAL METHODS QUALITY CONTROL INSTRUMENT AND EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE INSPECTION AND ACCEPTANCE OF SUPPLIES AND CONSUMABLES SAMPLE EQUIPMENT DECONTAMINATION DATA MANAGEMENT QUALITY OBJECTIVES AND CRITERIA OF MEASUREMENT SPECIAL TRAINING AND CERTIFICATIONS DOCUMENTATION AND RECORDS GPS AND SURVEY DATA	8 8 9 10 13 14 17 17 18 18 19 21 21 22
5	5.1	Quality assessment Internal audits of field activities Internal laboratory audit	22 22 23
6	6.1 6.2 6.3	VALIDATION AND USABILITY DATA REVIEW, VALIDATION, AND REPORTING LABORATORY DATA EVALUATION DATA DELIVERABLES DATA QUALITY ASSURANCE AND QUALITY CONTROL REVIEW DATA MANAGEMENT AND REDUCTION	24 24 24 26 27 27

LIMITATIONS

REFERENCES

**TABLES** 

## CONTENTS (CONTINUED)

**FIGURES** 

APPENDIX A

SUPPLEMENTAL LABORATORY INFORMATION: LABORATORY METHOD SUMMARIES

APPENDIX B

LABORATORY SURVEY RESULTS MEMORANDUM

APPENDIX C

**EXAMPLE CHAIN-OF-CUSTODY FORMS** 

## TABLES AND ILLUSTRATIONS

#### FOLLOWING PLAN:

#### **TABLES**

- 2-1 DISTRIBUTION LIST
- 2-2 CONTACT LIST
- 2-3 ANALYTICAL METHODS, PERFORMANCE CRITERIA, AND REPORTING LIMITS
- 3-1 PHSS ROD CULS, RALS, AND PTW THRESHOLDS FOR SEDIMENT
- 4-1 DATA QUALITY OBJECTIVES
- 4-2 SAMPLE CONTAINERS, METHODS, SAMPLE PRESERVATION, AND HOLDING TIMES
- 4-3 FIELD QUALITY CONTROL SAMPLE FREQUENCY
- 4-4 LABORATORY QUALITY CONTROL SAMPLE FREQUENCY

#### **FIGURES**

- 1-1 WILLAMETTE UPRIVER REACH BACKGROUND INVESTIGATION
- 2-1 ORGANIZATION CHART

## ACRONYMS AND ABBREVIATIONS

°C degrees Celsius

CRM certified reference material

DEQ Oregon Department of Environmental Quality

DGPS differential global positioning system

DQO data quality objective
EDD electronic data deliverable
EDL estimated detection limit

EMPC estimated maximum potential concentration EPA U.S. Environmental Protection Agency

FSP field sampling plan
HASP health and safety plan
LCS laboratory control sample

LCSD laboratory control sample duplicate

LDS laboratory duplicate sample LLOQ lower levels of quantitation MFA Maul Foster & Alongi, Inc.

MS matrix spike

MSD matrix spike duplicate

PHSS Portland Harbor Superfund Site PQL practical quantitation limit

QA/QC quality assurance and quality control

QAM quality assurance manager
QAPP quality assurance project plan
RB equipment rinsate blank
ROD record of decision

RPD relative percent difference

S2BVEM Stage 2B Validation Electronic and Manual S4VEM Stage 4 Validation Electronic and Manual

SOP standard operating procedure SRM sediment reference material

TOC total organic carbon

um micrometer

Upriver Reach Upriver Reach of the Lower Willamette River RMs 16.6

to 28.4

Work Plan sediment sampling work plan

## INTRODUCTION

This quality assurance project plan (QAPP) has been designed to support sediment investigation activities conducted in the Upriver Reach of the Lower Willamette River (Upriver Reach) (see Figure 1-1). The Upriver Reach is located between River Miles 16.6 and 28.4 of the Willamette River. This QAPP was prepared by Maul Foster & Alongi, Inc. (MFA) on behalf of the Oregon Department of Environmental Quality (DEQ) in cooperation with the U.S. Environmental Protection Agency (EPA). This QAPP is intended to support the sediment sampling work plan (Work Plan, to which this QAPP is an appendix), and the field sampling plan (FSP).

This QAPP was prepared to provide specific details on analytical methods and associated laboratory reporting and detection-limits and screening-level needs; and to define field and laboratory quality assurance and quality control (QA/QC) requirements and procedures as part of the background sediment investigation at the Upriver Reach.

#### 1.1 Quality Assurance Objectives

The objectives of this QAPP are to establish protocols to ensure that the data generated are of sufficient quality to support the data quality objectives (DQOs) and to ensure QA/QC protocols to maintain consistency of field and laboratory aspects of data collection and generation.

This QAPP was prepared using procedures consistent with the following EPA QAPP-specific guidance documents:

- EPA Requirements for Quality assurance Project Plans, QA/R-5, EPA/240/B-01/003 (EPA, 2001)
- Guidance for Quality Assurance Project Plans, QA/G-5, EPA/240/R-02/009 (EPA, 2002)
- Uniform Federal Policy for Quality Assurance Project Plans, Parts 1–3, EPA/505/B-04/900A through 900C (EPA, 2005)

This QAPP has been structured to reflect the format provided in the first two documents listed above as closely as possible. The following major sections of this QAPP correspond to the Groups prescribed in the QAPP guidance documents:

- The signature page, table of contents, and Sections 1 and 2, address Group A, Project Management.
- Sections 3 and 4 address Group B, Data Generation and Acquisition.
- Section 5 addresses Group C, Assessment and Oversight.

- Section 6 addresses Group D, Data Validation and Usability.
- References are provided at the end of this QAPP.

#### 1.2 Scope and Purpose

The purpose of this QAPP is to outline requirements and procedures for field sampling, field data collection, and laboratory analytical activities associated with the Upriver Reach investigation and to define the organizational structure of the cleanup program and the roles of individuals and subcontractors involved with the project.

This QAPP is designed to deliver:

- An investigation that meets project goals and achieves complete and accurate environmental data sets that have high precision and low bias.
- Environmental data that can be shown to be representative of actual Upriver Reach conditions.
- A QA/QC process allowing for comparability of environmental data sets so that the Upriver Reach can be characterized and assessed reliably.

# 2 project and task organization

## 2.1 Project Team Organization

This section provides the organizational structure, lines of authority, and responsibilities of key project roles. Project activities will be performed within the framework of the organization and functions presented in this section. The organizational structure will provide clear lines of responsibility and authority, with the following objectives:

- Identify appropriate lines of communication and coordination.
- Monitor project schedules and performance of subcontractors.
- Coordinate support functions such as laboratory analysis and data management.
- Provide progress QA reports.
- Provide corrective actions to rectify deficiencies.

The organizational structure for key project individuals with responsibilities for the sediment investigation is presented in Figure 2-1. An outline of the final report distribution is provided as Table 2-1. Contact information for key project individuals is provided in Table 2-2. The MFA project manager is responsible for distributing copies of the final QAPP to all individuals on the distribution list. The MFA QAM and the MFA project manager will provide QA status reports to all individuals

on the distribution list. Status reports will be issued by email on a weekly basis during field sampling and laboratory sample preparation and analysis.

#### 2.1.1 DEQ Project Manager Responsibilities

DEQ is the lead agency for the Upriver Reach. DEQ will oversee activities associated with the sediment sampling investigation as described in the Work Plan. DEQ will provide recommendations and guidance to the MFA project manager on conducting the sediment investigation in accordance with DEQ requirements. DEQ will review and approve the Work Plan and associated appendices, including this QAPP. The DEQ project manager is Mark Pugh.

### 2.1.2 EPA Project Manager Responsibilities

EPA is the lead agency for the Portland Harbor Superfund Site (PHSS), located downstream of the Upriver Reach. EPA will coordinate with DEQ to oversee the activities associated with the sediment sampling investigation as described in the Work Plan. EPA will review and approve the Work Plan and associated appendices, including this QAPP. Madi Novak is the EPA lead and remedial project manager. Benjamin Leake is the EPA remedial project manager assisting Ms. Novak.

#### 2.1.3 EPA Regional Quality Assurance Manager Responsibilities

The EPA Region 10 quality assurance manager (QAM) and is responsible for providing oversight and ensuring the implementation of the QC system outlined in this QAPP. The EPA QAM will be provided analytical data generated by the laboratories as well as any raw data that may support data validation. The EPA QAM will provide any comments to the DEQ Project Manager and will engage with MFA's QAM and Project Manager as necessary to address any significant issues. The EPA QAM, or delegated authority, will review and approve this QAPP. Donald M. Brown is the EPA QAM.

## 2.1.4 MFA Program Manager Responsibilities

The MFA program manager for the DEQ is Michael Pickering. He will assist the DEQ with overall programmatic planning for the technical and administrative components of the Upriver Reach investigation; oversee the development of scopes, schedules, and budgets; and administer the assignments via contracts with service providers.

### 2.1.5 MFA Project Manager Responsibilities

The investigation project manager is Josh Elliott. He will provide direction to other MFA staff and contracted service providers to complete the investigation in accordance with this QAPP. He will be responsible for all aspects of implementation of project-specific assignments and will report to the MFA program manager. The MFA project manager is the primary MFA point of contact with the DEQ and EPA.

### 2.1.6 Field Coordinator/On-Site Safety Officer Responsibilities

The field coordinators and on-site safety officers are Meaghan Pollock and Carolyn Wise. The field coordinators are responsible for leading investigation activities, including verifying that procedures for field activities are executed in the proper manner, activities are properly documented, the prescribed scope of work is completed, and communication protocols are met. Ms. Wise is an Oregon-registered geologist. The field investigation managers will report directly to the project manager. The on-site safety officers will also ensure that appropriate health and safety requirements associated with COVID-19 are in place during the field investigation as outlined in the health and safety plan (HASP), an appendix to the Work Plan.

#### 2.1.7 Project Scientist/Senior Technical Advisor

Phil Wiescher will be assigned as the project scientist and senior technical advisor. Additional MFA scientists or geologists will be assigned based on availability and relevant skills and experience. The scientists or geologists will work with the field team leaders and will be responsible for ensuring that investigation activities are conducted in accordance with this QAPP.

#### 2.1.8 Quality Assurance Manager Responsibilities

The MFA QAM is Erik Naylor. The MFA QAM will provide QA oversight for both the field sampling and laboratory programs, confirming that samples are collected and documented appropriately; coordinating with analytical laboratories; reviewing data quality; overseeing data validation; and supervising project QA coordination. The MFA QAM will report directly to the MFA project manager and will have a direct line of communication with the DEQ and EPA project managers and the database administrator. The MFA QAM will conduct internal peer reviews for agreement of validation decisions, assigned qualifiers, and changes made to the database by the project chemist/database administrator.

The MFA QAM and Project Manager will prepare the QA progress reports and provide to the EPA and DEQ project managers. QA progress reports will be provided by electronic mail on a weekly basis during field sampling activities and laboratory sample preparation and analysis.

### 2.1.9 Database Manager/Project Chemist Responsibilities

The MFA database administrator is Mary Benzinger. The database administrator oversees the management of analytics, field logs, spatial data, and other data transferred to or managed by MFA. The database administrator will report directly to the MFA project manager and the MFA QAM.

The project chemist is Mary Benzinger. The project chemist is responsible for validation of data generated during the Upriver Reach investigation. The data validator is responsible for performing independent reviews of the laboratory data packages for the data verification and validation criteria described in Section 6. The data validator is responsible for notifying the MFA QAM of issues related to quality or validity of laboratory procedures and laboratory data. When validation is complete, the

data validator will provide the database administrator with all applicable validated data, as described in Section 6.

#### 2.1.10 Procurement and Administrative Personnel

The MFA project manager, Josh Elliott, will be responsible for contract administration, including development and management of requests for proposals and bids and of contract documents for subcontractors providing services to DEQ. The contract administrator will be in close contact with the DEQ project manager (i.e., Mark Pugh).

#### 2.1.11 GIS Manager Responsibilities

Cascadia Associates will be responsible for the following:

- Preparing figures for the Work Plan and associated appendices
- Identification of sample locations, as described in the FSP

#### 2.2 Analytical Laboratory Responsibilities

The contracted laboratories are responsible for the following:

- Performing analyses based on methods described in this QAPP, including the methods referenced for each procedure
- Following documentation, custody, and sample logbook procedures
- Meeting reporting and QA/QC requirements
- Providing electronic data files as specified

The laboratory subcontractors are responsible for notifying the database administrator and the MFA QAM about issues relating to laboratory analysis. At the conclusion of work, the analytical laboratories will provide applicable analytical data, as described in Section 6, to the database administrator. Laboratories shall be certified to provide analytical laboratory services for the project-specific methods and matrices under the National Environmental Laboratory Accreditation Program, Oregon (including the Oregon Environmental Laboratory Accreditation Program).

Table 2-3 identifies the specific analytical methods, performance criteria, and reporting and detection limits that analytical laboratories are required to meet. Laboratory method summaries are provided in Appendix A; specific standard operating procedures are proprietary information which can be made available to EPA and DEQ project managers upon request.

The laboratory subcontractor responsible for dioxin/furan analysis will be Bureau Veritas Laboratories. Apex Laboratories, LLC will be responsible for grain size analysis and sieving samples to grain sizes required for analysis as described in the Work Plan. Pace Analytical Services will be the laboratory responsible for analysis of total organic carbon.

#### 2.3 Sampling Vessel Subcontractor Responsibilities

The sampling vessel subcontractor responsible for the sampling activities described in the final QAPP approved by EPA and DEQ will be Research Support Services of Poulsbo, Washington. Research Support Services will report directly to MFA.

# 3 PROJECT DESCRIPTION

## 3.1 Project Data Needs and Scope

As described in the Work Plan, there is uncertainty regarding the representativeness of the background-based record of decision (ROD) riverbank soil and sediment cleanup levels for dioxins/furans associated with the PHSS ROD cleanup levels. Limited dioxin/furan data were available in the Upriver Reach at the time CULs were developed. Subsequent sampling conducted in 2018 resulted in data biased towards finer-grained sediment.

The scope of this project includes establishment of unbiased background concentrations for dioxins/furans in the Upriver Reach from appropriate and feasible sample locations, evaluation of the influence of grain size on dioxin/furan concentrations, and the evaluation of whether potentially isolated, elevated concentrations of dioxins/furans are due to upstream source areas or anthropogenic background. The results of this investigation will support an updated evaluation of dioxin/furan background conditions in the Upriver Reach that will inform an evaluation of dioxin/furan soil and sediment cleanup levels established in the ROD.

## 3.2 Sample Program Design and Rationale

The sample program design and rationale for data collection activities are described below. More detailed information (i.e., proposed sample locations, sample collection procedures, and analytical scope) can be found in the Work Plan and its FSP appendix.

The following activities are applicable to this investigation:

• Identification of sample stations in areas of "medium" or "soft" river bottom substrate. Surface sediment sampling stations are selected using a stratified random grid system for areas classified as "medium" or "soft" river bottom substrate. The polygons span the river (center channel excluded) and sample points were randomly generated for placement in each polygon. The Upriver Reach river bottom substrate was previously classified into "soft", "medium", and "hard" substrate during a 2018 study using multiple lines of evidence, including acoustic sonar, mechanical hand probing, and visual classification. "Hard" river bottom substrates are not included in the sampling grid because these areas have been shown to result in sampling refusal.

- Attempts will be made to collect 45 three-point composite unbiased surface sediment samples. The number of sample locations was selected recognizing that sediment retrieval in some locations may be met with refusal despite targeting "medium" and "soft" river bottom substrates, and to enable collection of sufficient samples to support robust statistical analysis of background conditions.
- Collection of up to 45 three-point composite unbiased surface sediment samples including bulk sediment and two sediment grain-size fraction samples at each station (resulting in an estimated 135 unbiased samples). The following are the specific grain size fractions to be analyzed:
  - Sediment that passes a No. 10 sieve (<2,000 micrometers [um])</li>
  - Sediment that passes a No. 230 sieve (<62 um)

Analysis of sieved samples is being conducted to evaluate the potential influence of grain size on dioxin/furan concentrations. The grain size fractions were selected to differentiate between sediments that would be expected to remain bedded versus transported downstream.

• Analysis of sample for dioxins/furans, grain-size distribution, and total organic carbon (TOC) to support an evaluation of dioxin/furan concentrations and grain size.

#### 3.3 Parameters to Be Tested

Parameters to be tested in the laboratory are outlined below. Field parameters are detailed in the FSP, included as an appendix to the Work Plan. Further details regarding specific analytical methods, hold times, preservation requirements, laboratory reporting and detection limits, including estimated detection limits (EDLs), and practical quantitation levels or lower levels of quantitation (PQL/LLOQ) and units of measurement are provided in Section 4.

Laboratory-tested parameters for sediment samples include the following:

- Dioxins and furans
- TOC
- Grain-size distribution

Bulk sediment samples will be analyzed for TOC and dioxin/furans. Grain size fractions will also be analyzed for TOC and dioxin/furans following laboratory dry sieving. The specific grain size intervals for analysis of dioxin/furans and TOC include sediment that passes a No. 10 sieve (<2,000 um) and a No. 230 sieve (<62 um).

## 3.4 Project Schedule

Fieldwork is expected to begin in quarter 1 of 2021. The start date will depend on weather, subcontractor availability, and timing of scope approvals by the DEQ and EPA. The schedule related to sample preparation and analysis by the laboratory, and the subsequent reporting of the data will vary depending on the field work and the laboratory capacities at the time of analysis. Sample analysis

is anticipated to be completed during the second quarter of 2021 (see additional schedule details in Section 4.3 of the Work Plan).

## 4 DATA GENERATION AND ACQUISITION

### 4.1 Data Quality Objectives

The DQO process is used to establish performance and acceptance criteria, which serve as the basis for designing a plan for collecting data of sufficient quality and quantity to support the goals of the study (EPA, 2002). The seven steps of the DQO process are:

- 1. State the problem—define the problem, identify members of the planning team, and establish the schedule.
- 2. Identify the goal of the study—state how environmental data will be used to meet study objectives and solve the problem, identify study questions, and define alternative outcomes.
- 3. Identify information inputs—identify data and information needed to answer study questions.
- 4. Define the boundaries of the study—specify target population and characteristics of interest, define spatial and temporal limits, and define scale of inference.
- 5. Develop the analytic approach—define parameters of interest, specify type of inference, and develop logic for drawing conclusions from findings.
- 6. Specify performance or acceptance criteria—specify criteria for new data collection (performance metrics) and decision making (probability limits).
- 7. Develop the plan for obtaining data—develop the site-specific Work Plan including the QAPP and FSP.

The DQO process was used to develop this QAPP for environmental data collection. The specific DQOs developed for this project are provided in Table 4-1.

Concurrent with the development of this QAPP, a laboratory survey was conducted to identify laboratories that could best meet DQOs relevant to laboratory analyses (Appendix B).

## 4.2 Sample Process Design

## 4.2.1 Surface Sediment Sampling

Surface sediment sampling stations will be selected using a stratified random grid system. Sample stations will be collocated with varied grain sizes. See the FSP for additional details on surface sediment sampling.

#### 4.3 Sampling Methods

The FSP contains complete descriptions of the sample collection and handling methods. The types and numbers of samples that will be collected, the rationale for collection, and the analyses that will be performed are discussed in the FSP. The sections below provide a general description of the sampling methods.

#### 4.3.1 Nomenclature

#### 4.3.1.1 Surface Sediment Nomenclature

Sample containers will be labeled with an identification number that uniquely identifies the collected sediment sample. The sample nomenclature number will be logged in the field logbook or on the applicable sampling form as prescribed in the FSP:

- Sampling personnel
- Date and time of collection
- Field sample location and depth (as appropriate)
- Type of sampling (composite)
- Method of sampling
- Sampling matrix or source
- Intended analyses

The sample nomenclature for composite surface sediment samples will consist of up to four components: a three-letter project identification code, followed by a sample station code (consisting of a three-point composite), a sample matrix code, and a QC code (if appropriate). The following are examples of the nomenclature scheme:

- MFA-001-SS = MFA Upriver Reach background investigation, from station #1, surface sediment three-point composite
- MFA-010-SS = MFA Upriver Reach background investigation, from station #10, surface sediment three-point composite
- MFA-001-SS-DUP = MFA Upriver Reach background investigation, from station #1, surface sediment three-point composite, field duplicate sample

The sample nomenclature for surface sediment samples at specific grain size intervals will consist of a fifth component to the nomenclature above: a number indicating the specific sieve number associated with the sample fraction (if appropriate). This fifth component of nomenclature will be incorporated by the analytical laboratory. The following are examples of this additional nomenclature scheme:

• MFA-001-SS-10 = MFA Upriver Reach background investigation, from location #1, surface sediment three-point composite, passing through a No. 10 sieve (<2000 um)

• MFA-020-SS-230 = MFA Upriver Reach background investigation, from location #10, surface sediment three-point composite, passing through a No. 230 sieve (<62 um)

#### 4.3.1.2 Blanks Nomenclature

The sample nomenclature for the equipment rinsate blank (RB) samples will consist of three components: a three-letter project identification code, a QC code for the type of blank, and a sampling date.

The following are examples of the nomenclature scheme:

- MFA-RB-210112 = MFA Upriver Reach background investigation, rinsate blank collected January 12, 2021.
- MFA-RB-210215 = MFA Upriver Reach background investigation, rinsate blank, collected February 15, 2021.

If more than one RB is collected in a day, the time of collection will be added at the end of the sample name.

#### 4.3.2 Collection Methods

The sample collection methods, location control, field equipment, and decontamination procedures to be used are described in detail in the FSP.

### 4.3.3 Investigation-Derived Waste

Excess water or sediment remaining after sample processing will be returned to the vicinity of the collection site at the top of the water column. Any water or sediment spilled on the deck of the sampling vessel will be washed into the surface waters at the collection site before sampling proceeds to the next station.

All disposable materials used in sample processing, such as paper towels and disposable coveralls and gloves, will be placed in heavyweight garbage bags or other appropriate containers. Sampling personnel will remove disposable supplies from the Upriver Reach and place them in a normal refuse container for disposal at a solid waste landfill. Phosphate-free, detergent-bearing liquid wastes from decontamination of the sampling equipment will be washed overboard or disposed of the sanitary sewer system.

## 4.4 Sample Handling

#### 4.4.1 Hold Times

The first step in proper sample handling and custody is observance of analytical holding times, which can vary from 28 days to one year, depending on the media and analytical method(s) selected for the samples. Knowledge of required holding times will have a direct impact on the scheduling of sample

collection, packing, and shipping activities. The sample containers, volume, preservation, and holding times applicable to each analytical method are shown in Table 4-2.

#### 4.4.2 Sample Custody in the Field

Sample collection and sample custody procedures are designed so that field custody of samples is maintained and documented. These procedures provide identification and documentation of the sampling event and the sample chain of custody from shipment of sample bottle ware and precleaned sampling supplies, through sample collection, to receipt of the samples by the laboratory. When used in conjunction with the laboratory's custody procedures and documentation, these data establish full legal custody and allow complete tracking of a sample from preparation and receipt of sample bottle ware to sample collection, preservation, and shipping through laboratory receipt, sample analysis, and data validation. The chain of custody is defined as the sequence of persons who have the item in custody. Field custody procedures, sample packing, and shipping are described below. The persons responsible for sample custody, and a brief description of their duties, are as follows:

- Laboratory Sample Custodian or Commercial Supplier: Verifies that the bottle ware is certified clean; arranges for bottle ware shipment to field sampling personnel.
- **Field Staff:** Receives the sample bottle ware from the laboratory, inspects the bottle ware for physical integrity; retains the shipping invoice or packing list from the shipping courier as documentation of the transfer of the bottle ware; collects and preserves samples; retains the bottle ware and samples under custody until sample shipment; relinquishes samples to the shipping courier or the laboratory representative.
- Laboratory Project Manager: Verifies reported laboratory analyses to the sample chain-of-custody record; assures that chain-of-custody documentation is incorporated into the project file.

A sample or other physical evidence is in custody if it is or was:

- In the actual possession of the field investigator, transferee, or laboratory technician.
- In the view of the field investigator, transferee, or laboratory technician after being in their physical possession.
- In the physical possession of the field investigator, transferee, or laboratory technician and then secured by them to prevent tampering.
- Placed in a designated secure area.

## 4.4.3 Chain-of-Custody Record

The field chain-of-custody record is used to record the custody of samples or other physical evidence collected and maintained. This form will not be used to document the collection of split or duplicate samples. The chain-of-custody record also serves as a sample logging mechanism for the analytical laboratories' sample custodians.

The following information must be supplied in the indicated spaces in detail to complete the field chain-of-custody record:

- Project-specific information, including the project number and project name.
- The signature of the sampler and/or the sampling team leader in the designated signature block.
- The sampling location identification number, date and time of sample collection, grab or composite sample designation, and sample preservation type must be included on each line (each line should contain only those samples collected at a specific location).
- The total number of sample containers must be listed in the indicated space for each sample, and the total number of individual containers must also be listed for each type of analysis.
- The field investigator and subsequent transferee(s) must document the transfer of the samples listed on the chain-of-custody record in the spaces provided at the bottom of the form. Both the person relinquishing the samples and the person receiving them must sign the form and provide the date and time of transfer in the proper space on the form. Usually, the last person receiving the samples or evidence should be a laboratory sample custodian.

The chain-of-custody record is a serialized document; once it is completed, it becomes an accountable document and must be maintained in the project file. The suitability of any other form for chain of custody should be evaluated based on its inclusion of the above information in a legible format. Examples of chain-of-custody records for each laboratory described in this document are provided as Appendix C.

## 4.4.4 Sample Packaging and Shipment

Samples are packed for shipping in waterproof ice chests and coolers. Depending on container type, the sample containers may be individually sealed in Ziploc® or similar plastic bags before they are packed in the cooler with bubble wrap or Styrofoam packing. Wet ice will be double-bagged in plastic bags (to inhibit cross-contamination of samples by melt water) and placed with the samples in the cooler to maintain the samples at a temperature of 0 to less than 6 degrees Celsius (°C) during shipping.

The chain-of-custody record that identifies the samples is signed as "relinquished" by the principal sampler or responsible party. This chain-of-custody record is sealed in a waterproof plastic bag and is placed inside the cooler, typically by taping the bag to the inside lid of the cooler.

Following packing, the cooler lid is sealed with packing tape. A custody seal is signed, dated, and affixed from the cooler lid to the cooler body, and is also covered with clear tape. This ensures that tampering with the cooler contents will be immediately evident.

The sample coolers will be shipped by courier to the laboratory in accordance with laboratory schedule requirements. A copy of the shipping invoice is retained by the field manager and becomes part of the sample custody documentation.

#### 4.5 Laboratory Procedures

The analytical laboratories named in this QAPP have established programs of sample custody that are designed to ensure that each sample is accounted for at all times. The objectives of these sample custody programs include the following:

- Unique identification of the samples, as appropriate for the data required
- Analysis of the correct samples and traceability to the appropriate record
- Preservation of sample characteristics
- Protection of samples from loss or damage
- Documentation of any sample alteration (e.g., filtration, preservation)
- Establishing a record of sample integrity for legal purposes

The standard operating procedures (SOPs) for sample custody protocol are maintained by the laboratories and adhered to by laboratory personnel. The sample custody SOPs are in the laboratories' SOP libraries and/or QA manuals.

#### 4.5.1 Intra- and Interlaboratory Sample Transfer

The laboratory project manager will ensure that a sample-tracking record that follows each sample through all stages of laboratory processing is maintained. This record must contain, at a minimum, the names of individuals responsible for performing the analysis; the dates of sample extraction, preparation, and analysis; and the type of analysis being performed.

Any sample, homogenate, or sample extract that will need further analysis that will not be performed by the initial contracted laboratory and that requires inter- or intralaboratory transfer will be subject to all specifications described in the previous section. Sample analyses, as shown in Table 2-3, will not be subcontracted to outside laboratories or transferred to other laboratories within the specific laboratory organization without consultation with the MFA project manager, DEQ, and EPA.

## 4.5.2 Archived Samples

All excess sediment samples submitted to the analytical laboratory will be archived at less than -10°C. It is not expected that material will be archived for future grain size analysis; however, if such material is archived, it will not be frozen. The laboratories will maintain chain-of-custody documentation and proper storage conditions for the entire time that the samples are in their possession. All laboratories for this project will store the excess samples for up to 12 months following completion of data validation. The laboratories will not dispose of the samples for this project until they are authorized to do so by the MFA QAM, DEQ, and/or EPA.

#### 4.6 Analytical Methods

The selected analytical methods, performance criteria, and laboratory reporting limits summarized in Table 2-3 were selected to meet the data needs and scope summarized in Table 4-1.

Analytical methods include:

- Dioxins and furans by EPA Method 1613B
- TOC by EPA 9060A
- Grain size distribution by ASTM D-422 modified (the modification to the original method includes an additional sieve, no. 230)

#### 4.7 Quality Control

The quality of data will be monitored and verified by maintaining logs, documenting field activities, and collection and analysis of field and laboratory QC samples. Tables 4-3 and 4-4 provide a summary of the field and laboratory QC samples, along with the required collection frequency and acceptance criteria for each sample matrix and QC type.

#### 4.7.1 Field Quality Control Samples

Field QC samples are used to assess the accuracy and precision of the field sample collection and handling activities (see Table 4-3). Field QC samples will be analyzed for the same parameters as the investigative samples, with the exception of grain size. The results of the field QC samples described below will be reported along with investigative sampling results when applicable.

#### 4.7.1.1 Equipment Blanks

Analyses of RBs will be used to assess the efficiency of sampling equipment decontamination procedures in preventing cross-contamination of samples. RBs will be collected by pouring new (produced three months prior or newer) laboratory-certified distilled or deionized water over or through decontaminated (clean) sampling equipment used in the collection of investigative samples and subsequently collected in prepared sampling containers. Laboratory-certified distilled or deionized water will be provided by the same laboratory that is analyzing the RB samples. The RBs will be shipped with the associated field samples.

RBs must be collected from tools used to collect samples, such as spoons and bowls and the power grab sampler. RBs are not required for dedicated, disposable equipment used for sampling (e.g., plastic spoons; gloves). One RB will be collected and submitted to the analytical laboratory for each day on which samples are collected.

#### 4.7.1.2 Trip Blanks

Trip blanks are not planned for this study because the field samples will not be analyzed for volatiles.

#### 4.7.1.3 Field Duplicates

Field duplicate samples are collected to assess reproducibility of field procedures. For nonaqueous matrices, sample heterogeneity may affect the measured precision for the duplicate sample. Field duplicate sample collection will follow the same procedures used to collect investigative samples. The duplicate samples will be collected from the same location and depth interval as the parent-investigative sample immediately after the parent-investigative sample is collected. Two sets of samples from a single source are prepared, labeled with unique sample numbers, and submitted to the laboratory. One field duplicate will be prepared for every 20 environmental samples collected (see Table 4-3).

Field replicates (unique samples processed from collocated stations in the field) are not planned for this study because the field samples are composites.

#### 4.7.2 Laboratory Quality Control Samples

The laboratory QC samples will be used to assess the accuracy and precision of the laboratory sample collection and handling activities. The results for the QC samples described below will be reported along with investigative sampling results when applicable to the analytical method used. Types and frequencies of analytical QC samples are shown in Table 4-4.

#### 4.7.2.1 Calibration Verification

Instruments will be calibrated prior to sample analysis according to method requirements and when any ongoing calibration does not meet calibration verification criteria. The number of points used in the initial calibration is defined in the analytical method. Calibration will be continued as specified in the analytical method to track instrument performance. If a continuing calibration does not meet control limits, analysis of project samples will be suspended until the source of the control failure is either eliminated or reduced to within control specifications. Any project samples analyzed while the instrument was outside control limits will be reanalyzed.

#### 4.7.2.2 Matrix Spike/Matrix Spike Duplicate

Matrix spike/matrix spike duplicate (MS/MSD) samples are analyzed to assess the matrix effects on the accuracy of analytical measurements. The methods required for conducting the work associated with this QAPP do not typically include analysis of MS/MSD samples. In the case of EPA Method 1613B, each sample is spiked with a labeled standard that serves a purpose similar to that of an MS.

#### 4.7.2.3 Labeled Standards

Labeled analogs and surrogate spiking consist of adding reference compounds to samples before sample preparation for EPA Method 1613B. Labeled standards are used to assess method accuracy on a sample-specific basis. Labeled analogs will be added to each sample in accordance with the analytical method requirements.

#### 4.7.2.4 Method Blanks

Method blanks are prepared using analyte-free (reagent) water and are processed with the same methodology (e.g., extraction, digestion) as the associated investigative samples. Method blanks are used to document contamination resulting in the laboratory from the analytical process. One method blank in every analytical batch shall be prepared and analyzed.

The method blank results are used to verify that reagents and preparation do not impart unacceptable bias to the investigative sample results. The presence of analytes in the method blank sample will be evaluated against method-specific thresholds. If analytes are present in the method blank above the PQL/LLOQs, corrective action will be taken to eliminate the source of contamination before analysis proceeds. Investigative samples of an analytical batch associated with method blank results outside acceptance limits will be qualified as appropriate by the MFA QAM or database manager.

#### 4.7.2.5 Laboratory Control Samples

Laboratory control samples (LCSs) and laboratory control sample duplicates (LCSDs) are prepared by spiking laboratory-certified, reagent-grade water or sand blanks with the analytes of interest or a certified reference material that has been prepared and analyzed. The result for percent recovery and relative percent difference (RPD) of the LCS/LCSD is a data quality indicator of the accuracy and reproducibility of the analytical method and laboratory performance. LCS/LCSDs shall be prepared and analyzed in every analytical batch.

#### 4.7.2.6 Laboratory Duplicate Samples

Laboratory duplicate samples (LDSs) are prepared by the laboratory by splitting an investigative sample into two separate aliquots and performing separate sample preparation and analysis on each aliquot. The results for RPD of the primary investigative sample and the respective LDSs are used to measure precision in the analytical method and laboratory performance. For nonaqueous matrices, sample heterogeneity may affect the measured precision for the LDS. LDSs shall be prepared and analyzed for every analytical batch.

#### 4.7.2.7 Laboratory Split Samples

The split sampling process, if completed, will be described in a Split Sampling QAPP provided by the requesting agency.

#### 4.7.2.8 Sediment Reference Material

One sediment reference material (SRM) sample, Puget Sound SRM, will be requested and analyzed once for dioxins and furans. The SRM sample is matrix-specific, with known concentrations of dioxins and furans. The SRM will be assessed by comparing laboratory results to the certified performance criteria. The results will be validated and reported per the U.S. Army Corps of Engineers requirements (COE, 2016).

Up to eight certified reference material (CRM) samples, Cerilliant brand CRM, will be requested and analyzed for dioxin and furans. One CRM sample will be analyzed per batch of samples submitted to the analytical laboratory. The CRM sample (Cerilliant catalog number EDF-5183) contains low-level concentrations of dioxins and furans. The CRM will be assessed by comparing laboratory results to the certified performance criteria. The results will be validated and reported per the U.S. Army Corps of Engineers requirements (COE, 2016).

#### 4.8 Instrument and Equipment Testing, Inspection, and Maintenance

Instruments for field parameter measurements will follow the sampling and analysis plan protocol and manufacturers' recommendations for testing, inspection, and maintenance. Field equipment used for obtaining samples will be decontaminated as required and stored in a clean and secure location.

Instruments for field parameter measurements will follow the Work Plan and FSP requirements and manufacturers' recommendations for calibration. Instruments will be calibrated at the beginning of each sampling event. Calibration checks will be conducted at the beginning of each sampling day. Calibration may be conducted again during a sampling event, as necessary, based on the results of the calibration check. Calibration records will be recorded in the field logbooks.

Laboratory instruments and equipment will comply with the contracted laboratories' QA/QC procedures for testing, inspection, and maintenance. Laboratory instrument and equipment testing, inspection, and maintenance documentation will be provided to the MFA QAM if requested.

Preventative maintenance in the laboratory will be the responsibility of the laboratory personnel and analysts. At a minimum, the preventative maintenance schedules contained in the EPA methods and laboratory SOPs and in the equipment manufacturers' instructions will be followed. This maintenance includes routine care and cleaning of instruments, and inspection and monitoring of the carrier gases, reagents, solvents, reference materials, and glassware used in analysis. All maintenance of instruments and procedures will be documented in maintenance log/record books. Each of the laboratories has included SOPs for preventative maintenance in their individual QA manuals.

### 4.9 Inspection and Acceptance of Supplies and Consumables

Sample container requirements are shown in Table 4-2. Other supplies include, but are not limited to, deionized water, chemicals for decontamination, sample collection equipment, and personal protective equipment. All will be obtained from reputable suppliers with appropriate documentation or certification. Supplies will be inspected to confirm that they meet use requirements, and certification records will be kept in project files. If contamination is visible in materials, the materials will be discarded. Nondedicated field equipment will be decontaminated prior to use in accordance with Section 4.3.3.

The analytical laboratory will inspect supplies and consumables before their use in analysis. The materials description in the analytical methods will be used as a guideline for establishing acceptance criteria. Purity of reagents will be evaluated through analysis of LCSs and method blank samples. The

laboratory shall maintain an inventory of supplies and consumables. No materials will be used after the manufacturers' expiration dates.

#### 4.10 Sample Equipment Decontamination

Sampling equipment and reusable materials that contact sample media will be decontaminated between uses per the procedures in the FSP. Decontamination will generally involve the following:

- Rinse the equipment with potable water to remove visible soil, petroleum sheen, or contamination.
- Scrub the equipment with a brush and solution of distilled water and non-phosphate detergent.
- Rinse the equipment with distilled water.
- Allow equipment to air dry or use paper towels.
- At all times, ensure the decontaminated equipment is stored in a manner the prevents it from becoming contaminated while not in use. Depending on the size of the equipment, it can be wrapped with new aluminum foil or placed in a new plastic bag.

#### 4.11 Data Management

#### 4.11.1 Field Logbooks and Forms

Field investigation personnel will be responsible for maintaining a daily record of significant events, observations, and measurements during field investigations. Field records may consist of a bound logbook or paper or electronic field data sheets. A separate entry will be made for each sample collected. Specific field recording procedures will be identified in the FSP as needed. Field logbooks and forms will be included in the project files at the end of field activities to provide a record of sampling.

## 4.11.2 Electronic Data Management

The laboratory shall record the results of each analysis in a Laboratory Information Management System in accordance with the contracted laboratory's quality assurance plan. Data will be provided to MFA as electronic data deliverables (EDDs), which will be imported directly into an EQuIS database used for data storage. Validated laboratory results will be exported and provided as part of the final report for each project.

Data will be managed in such a way that they can be provided to EPA and DEQ in a standardized database schema for electronic data reporting and delivery with data reports to EPA Region 10 and DEQ.

## 4.12 Quality Objectives and Criteria of Measurement

#### 4.12.1 Precision

Precision is the measure of agreement among repeated measurements of the same property under identical or substantially similar conditions, calculated as either the range or the standard deviation (EPA, 2002). Precision is measured by making repeated analyses on the same analytical instrument (LDSs) or replicate collections of samples in the field (field duplicates that are not incremental sample methodology replicate samples). Sampling precision will be measured through the laboratory analysis of field duplicate samples. Laboratory precision will be measured through the analysis of LCS/LCSD pairs and laboratory duplicate pairs.

Precision criteria are expressed as the RPD between the primary and duplicate samples. The acceptance limits for RPD are based on the sample matrix and the analytical method used.

The RPD is calculated using the equation:

$$RPD = \frac{|x_s - x_d|}{(x_s + x_d)/2} \times 100\%$$

Where:

 $x_s$  = result for primary sample  $x_d$  = result for duplicate sample

For field duplicates, the precision goal for this project is RPD of 50 percent. For laboratory duplicates (chemistry), the precision goal for this project is 35 percent. Non-detects and results at or near the PQL/LLOQ will be considered in the interpretation of data precision.

### 4.12.2 Accuracy and Bias

Accuracy is defined as the measure of the overall agreement of a measurement with a known value and includes a combination of random error (precision) and systematic error (bias) components of both sampling and analytical operations (EPA, 2002). Inasmuch as the "true" concentration of sampled media is not known, the degree of accuracy in the measurement is inferred from recovery data determined by sample spiking and/or the analyses of reference standards. The criterion for accuracy is expressed as the percent recovery of the sample spiking. The acceptance limits for percent recovery are based on the analytical method used.

Bias is defined as the systematic or persistent distortion of a measurement process that causes error in one direction (EPA, 2002). Data bias is addressed in the field and the laboratory by calibrating equipment, through collection and analysis of QC blank samples, and through the analysis of QC standard samples.

Accuracy, when potentially affected by high or low recoveries, is presented as percent recovery. Percent recovery is calculated using the equation:

Percent Recovery = 
$$\frac{x_{SS} - x_{S}}{T} \times 100\%$$

Where:

 $x_{ss}$  = spiked sample concentration

 $x_s$  = sample concentration

T = true value of added spike (spike concentration)

Accuracy goals based on typical laboratory control limits for analytical results are presented as upper and lower control limits for LCS and LCSD percent recovery in Table 2-3. Method-based control limits will be used for the accuracy goals for labeled analogs.

### 4.12.3 Completeness

Data completeness is defined as a measure of the amount of valid data needed from a measurement system (EPA, 2002). The completeness goal is essentially the same for data uses in that sufficient amounts of valid data are to be generated. The percent completeness for each set of samples will be calculated as follows:

$$\% \ Completeness = \frac{Valid\ Data}{Total\ Data\ Collected} \times 100$$

The QA objective for completeness for the parameters will be 90 percent.

## 4.12.4 Representativeness

Data representativeness is a qualitative term that expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition (EPA, 2002). Data representativeness is evaluated by assessing the accuracy and precision of the sampling program.

Representativeness is ensured by collecting sufficient numbers of samples of an environmental medium, properly chosen with respect to place and time. The precision of a representative set of samples reflects the degree of variability of the sampled medium as well as the effectiveness of the sampling techniques and laboratory analysis. A statistically robust and valid sampling program that will achieve the objectives of the study was developed. The statistical basis for development of the sediment scope is described in Section 2.1 of the FSP. Based on these analyses and number of samples, the surface sediment samples are expected to represent current conditions within the Upriver Reach. The criterion for evaluating representativeness will be satisfied by confirming that the sample collection procedures are consistently followed as outlined in the Work Plan, the FSP, and this QAPP.

#### 4.12.5 Comparability

Data comparability is a qualitative term that expresses the measure of confidence with which one data set can be compared to another and can be combined with other data sets for decision making (EPA, 2002). Data comparability will be achieved by using standard sampling and operating procedures and analytical methods. Comparable data sets must contain the same variables of interest and must possess values that can be converted to a common unit of measurement. Comparability is normally a qualitative parameter that is dependent on other data quality elements. For example, if the detection limits for a target analyte were significantly different for two different methods, the two methods would not be comparable. Using standard sampling and analytical procedures, and carefully assessing laboratory capabilities, will increase the likelihood that data sets will be comparable by individual entities. Data comparability will be assessed through documentation of QA/QC procedures.

#### 4.12.6 Sensitivity

Data sensitivity is defined as the capability of a method or instrument to discriminate between measurement responses representing different levels of the variable of interest (EPA, 2002). Analytical sensitivity is readily evaluated by comparing laboratory reporting and detection limits to PHSS ROD cleanup levels and risk-based screening values (see Table 3-1). The reporting and detection limits specified through the DQO process are based on Bureau Veritas Laboratories' limits (see Table 2-3). Results measured between the PQL/LLOQ and the EDLs will be reported for all analytes and assigned the appropriate qualifier.

## 4.13 Special Training and Certifications

All project personnel performing work at the Upriver Reach will be health and safety trained as specified in the HASP, included as an appendix to the Work Plan. The HASP describes the specialized field training and certification required for personnel and provides documentation of this training. The MFA project manager is responsible for ensuring all project personnel have the appropriate training to perform the activities outlined in this QAPP.

Laboratories shall be certified to provide analytical laboratory services for the project-specific methods and matrices under the National Environmental Laboratory Accreditation Program (including the Oregon Environmental Laboratory Accreditation Program) (see Appendix B).

#### 4.14 Documentation and Records

Field investigation personnel will be responsible for maintaining a bound field notebook in order to provide a daily record of significant events, observations, and measurements during field investigations. All entries will be signed and dated. A separate entry will be made for each sample collected. Field logbooks and forms will be transferred to the MFA project manager's project files at the end of field activities to provide a record of sampling. Electronic forms may also be used to record the information described above. Copies of the field logbooks and forms will be made available to the DEQ and EPA.

Field investigation personnel will be responsible for maintaining an equipment calibration log to record the calibration measurements and the frequencies of equipment calibration. This log may be incorporated into the field logbook notes for a specific date and activity.

All project-related information will be routed to the MFA project manager, who will be responsible for distributing the information to appropriate personnel. The official project files will be maintained in MFA's Portland, Oregon, office. Project documentation will be archived for a minimum of 20 years after completion of the investigation.

#### 4.15 GPS and Survey Data

A differential global positioning system (DGPS) will be used to locate the sampling position for each proposed location outlined in the FSP. Sampling locations will be determined to an accuracy of ±3 meters. Horizontal coordinates will be referenced to the Washington South State Plane HARN (NAD83). Effort will be made to collect sediment from each location; however, some locations may remain inaccessible. Samples may be field adjusted and will be collected as close as possible to the intended sample location. The DGPS will be used to record each location that has been field adjusted. Locations may be accessed by boat or on foot (e.g., locations adjacent to the shoreline).

# 5 DATA QUALITY ASSESSMENT

The project QA/QC manager or their designee may conduct both performance and systems audits of field and laboratory activities, as necessary. This section discusses the types of audits, including system audits of the field and laboratory prior to project startup and ongoing field and laboratory performance audits over the course of project activities.

#### 5.1 Internal Audits of Field Activities

The field systems audit is an on-site audit that focuses on data-collection systems, using this QAPP as a reference. Specific activities vary with the scope of the audit, but can include a review of the following:

- Sample-collection activities
- Equipment calibration techniques and records
- Decontamination and equipment cleaning
- Background and training of personnel
- Sample containers and preservation techniques
- Chain-of-custody procedures

Performance audits of field screening and sampling activities will be conducted using a review of field logbooks, field sampling sheets, chain-of-custody forms, health and safety sign-in sheet(s), laboratory sample receipt forms, and in person documentation of field procedures by the MFA Project Manager or MFA QAM. The need for a field systems audit will be determined by the MFA Project Manager in coordination with the MFA QAM. If the field system audit is conducted, it would occur at a frequency of no more than one field system audit per week.

An inspection for suitability of the samples for proper laboratory analysis serves as the performance audit of the sample collection procedures. For example, insufficient sample volume for analysis, or improper preservation of samples will be noted by the analytical laboratory. A preponderance of such reports of unsuitable samples will indicate that the sampling procedures are poor or unacceptable. Analytical results will be reviewed by the MFA project manager and the MFA QAM manager to assess the performance and adequacy of sample collection procedures.

#### 5.2 Internal Laboratory Audit

The laboratory systems audit is a review of laboratory operations to verify that the laboratory has the necessary facilities, equipment, staff, and procedures in place to generate acceptable data.

Specific activities vary with the scope of the audit, but can include a review of the following:

- Equipment suitability and maintenance/repair
- Background and training of personnel
- Laboratory control charts and support systems
- QA samples, including performance evaluation samples
- Chain-of-custody procedures
- Data logs, data transfer, data reduction, and validation

The project laboratories participate in a variety of federal and state programs that subject laboratories to stringent performance audits on a regular basis. QA policies and procedures currently in place at the laboratories, and actions that will be included in sampling activities to ensure QA conformance, include the following:

- Both intra- and interlaboratory check samples
- Periodic (at least annual) audits conducted by the corporate QA office
- LCSs prepared and/or analyzed as applicable to the method(s) at a frequency equal to at least 5 percent of the total number of samples analyzed

Laboratory performance will be monitored by the MFA QAM. If necessary, or at the request of the DEQ or EPA project manager, the MFA QAM will conduct an on-site audit of field operations or any of these laboratories at a frequency of no more than once per year per laboratory or once per major sampling event.

When a problem situation arises regarding any significant impediment to the progress of the investigation, corrective action will be implemented to identify the problem and its source. Appropriate documentation of this action will be recorded in the project file.

Personnel responsible for the initiation and approval of a corrective action will be the laboratory QA manager (for a corrective action at the laboratory) and the MFA QAM (for corrective actions identified during field activities and/or during the data validation effort). The MFA project manager will be responsible for the approval of corrective action measures.

## 6 DATA VALIDATION AND USABILITY

This section describes the stages of data quality assessment after data have been received. It addresses data reduction, review, verification, and validation. It also discusses the procedures for evaluating the usability of data with respect to the DQOs set forth in Section 4.

#### 6.1 Data Review, Validation, and Reporting

Data validation is the process of evaluating the completeness, correctness, and compliance of a specific data set against the method, procedural, or contractual specifications (EPA, 2002). Data validation is confirmation by examination and provision of objective evidence that the particular requirements for specific intended use have been fulfilled (EPA, 2001) and is an analyte- and sample-specific process that extends the evaluation of data beyond method, procedural, or contractual compliance (i.e., data verification) to the analytical quality of a specific data set (EPA, 2002).

The MFA QAM or database manager will be responsible for ensuring that the necessary reporting components have been included in laboratory reports, such as necessary fields (e.g., collection/analysis dates, units) as well as the presence of (but not implications of) QA/QC data components (e.g., LCS records, surrogate results). The MFA QAM or database manager will also verify that all field samples are reported, that the correct analytical methods were used, and that the requested target analytes were reported. For samples associated with this project, the laboratory will produce data packages that will contain the information needed for validation of the data, as described in Section 4.15. Additional detail on laboratory data evaluation, including verification and validation, is provided in the sections below.

### 6.2 Laboratory Data Evaluation

## 6.2.1 Laboratory Data Verification Methods

The laboratory analyst will be responsible for the reduction of raw data generated at the laboratory bench and verification that data reduction performed by the laboratory instrument or Laboratory Information Management System is correct.

QC checks for data verification that will be performed for all generated data are as follows:

- Verify that batch QC and field samples were analyzed at the specified frequency.
- Verify compliance of calibrations and calibration checks with laboratory criteria.
- Verify that holding times for extraction and analyses and sample preservation were met.
- Verify that the EDLs and PQL/LLOQs were met.
- Verify that all project and QC sample results were properly reported and flagged.
- Review chain-of-custody documentation to verify completeness of the sample set for each data package submitted.
- Assess the impact of laboratory and field QC results.

These QC checks will be performed by laboratory analysts, the assigned laboratory project manager or supervisor, laboratory QC specialists, or a combination of these personnel. After the data reports have been reviewed and verified, the laboratory reports will be signed and released for distribution.

#### 6.2.2 Laboratory Data Validation Methods

Validation of the analytical data produced under this QAPP will be performed by the MFA QAM and the project chemist (see Section 2). The personnel will review laboratory performance criteria and sample-specific criteria. All validation reports and corresponding laboratory reports will be made available with the final project report and upon EPA's request.

Validation will be performed for 100 percent of the data report packages for each applicable analysis type. The data validation review will include review of the following items from the Stage 2B Validation Electronic and Manual (S2BVEM) laboratory data reports: consistency with the chain of custody; holding times; labeled analog recoveries; field duplicate agreement; laboratory duplicate precision; LCS/LCSD accuracy and precision; RBs; and method blank analyses, consistent with EPA S2BVEM level data validation and verification requirements (EPA, 2009).

For a minimum of 10 percent of the data, the MFA QAM or the project chemist will review Stage 4 Validation Electronic and Manual (S4VEM) data packages to assess laboratory performance criteria (e.g., initial calibration, continuing calibration, tuning, sensitivity, degradation), chromatograms, and calculation checks (EPA, 2009). If Stage 4 validation reveals data quality issues that Stage 2 validation cannot identify, Stage 4 validation may be considered for the entire data set. Sample reanalysis may be trigged due to data quality issues. The QC elements to be reviewed for the full data validation include the following for organic analyses:

Organic analyses (dioxins/furans and TOC):

- Consistency with chain of custody
- Holding times
- Initial calibration

- Continuing calibration
- Blanks
- Labeled analog recovery
- LCS/LCSD recovery and RPD
- LDS RPD
- Internal standard performance
- Field duplicate sample analysis RPD
- Compound identification
- Compound quantitation and detection limits
- Recalculations from the raw data
- Estimated maximum potential concentrations (EMPCs)
- System performance
- Overall assessment of the data

Data validation reports will provide the appropriate data validation label (i.e., S2BVEM or S4VEM). The data validator (MFA QAM) will review data and assign data qualifiers to sample results, following EPA guidelines and/or as determined in coordination with DEQ and EPA when assigning qualifiers, including EMPCs (EPA, 2016, 2017).

Data qualifiers are used to classify sample data as to their conformance to QC requirements. The most common qualifiers are:

- J—Estimate, qualitatively correct but quantitatively suspect.
- R—Reject, data not suitable for any purpose.
- U—Not detected at a specified detection limit.
- UJ—Not detected at a specified detection limit and an estimated value, qualitatively correct but quantitatively suspect

Poor surrogate, blank contamination, or calibration problems, among other things, can require qualification of the sample data. The reasons for the qualifications will be stated in the data validation report.

#### 6.3 Data Deliverables

Standard (Tier II) laboratory data deliverables will include:

- Transmittal cover letter
- Case narrative
- Analytical results
- EDLs and PQL/LLOQs
- Laboratory qualifiers, including EMPCs
- Chain of custody
- Surrogate, labeled analogue, and internal standard recoveries

- Method blank results
- LCS/LCSD results
- Laboratory duplicate results
- EDD

In addition to the Tier II deliverable list, Tier IV laboratory deliverables, including the following, will be provided as requested:

- All the pertinent standards information and traceability and standard logbook information for individual standard solutions
- All the pertinent calibration data and continuing calibration data, including tune information
- All the raw data chromatograms and instrument printouts for the sample results and calibration data
- Internal standard area and retention time summaries
- All the pertinent sample preparation information
- Preparation batch and analytical batch associations
- Before-and-after manual integration chromatograms
- Any correspondence between the laboratory and the client regarding sample issues

## 6.4 Data Quality Assurance and Quality Control Review

As described above, the MFA QAM and the project chemist will assess the laboratory analytical data to determine their usability in relation to the DQOs and confirm whether the DQOs are being met. The findings of the data validation will be documented in a data validation report, which will be provided to the MFA project manager and the program manager; it will also be included, along with the qualified data, in the final project report submitted to the DEQ and EPA.

### 6.5 Data Management and Reduction

MFA uses EQuIS to manage all laboratory data. The laboratory will provide the analytical results in electronic EQuIS-deliverable format. After data evaluation, data qualifiers will be entered into the EQuIS database.

Data may be reduced to summarize particular data sets and to aid interpretation of the results. Statistical analyses may also be applied to results. Data-reduction QC checks will be performed on all hand-entered data, any calculations, and any data graphically displayed. Data may be further reduced and managed using one or more of the following computer software applications:

- Microsoft Excel® (spreadsheet)
- EQuIS (database)

- AutoCAD® and/or ArcGIS (graphics)
- EPA ProUCL (statistical software)

## LIMITATIONS

The services undertaken in completing this plan were performed consistent with generally accepted professional consulting principles and practices. No other warranty, express or implied, is made. These services were performed consistent with our agreement with our client. This plan is solely for the use and information of our client unless otherwise noted. Any reliance on this plan by a third party is at such party's sole risk.

Opinions and recommendations contained in this plan apply to conditions existing when services were performed and are intended only for the client, purposes, locations, time frames, and project parameters indicated. We are not responsible for the impacts of any changes in environmental standards, practices, or regulations subsequent to performance of services. We do not warrant the accuracy of information supplied by others, or the use of segregated portions of this plan.

COE. 2016. Guidance for SRM distribution and reporting. U.S. Army Corps of Engineers. March 16.

EPA. 2001. EPA requirements for quality assurance project plans, EPA QA/R-5. EPA/240/B-01/003. U.S. Environmental Protection Agency. March. (reissued May 2006).

EPA. 2002. EPA guidance for quality assurance project plans, EPA QA/G-5. EPA/240/R-02/009. U.S. Environmental Protection Agency. December.

EPA. 2005. Uniform federal policy for quality assurance project plans. Part 1 UFP-QAPP manual, Intergovernmental Data Quality Task Force. Vers. 1. March. EPA-505-B-04-900A. U.S. Environmental Protection Agency. March.

EPA. 2009. Guidance for labeling externally validated laboratory analytical data for Superfund use. EPA 540/R-08/005. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response. January.

EPA. 2016. EPA Superfund contract laboratory program, national functional guidelines for high resolution Superfund methods data review. EPA 542-B-16-001. U.S. Environmental Protection Agency, Office of Superfund Remediation and Technology Innovation. April.

EPA. 2017. EPA contract laboratory program, national functional guidelines for Superfund organic methods data review. EPA 540-R-2017-002. U.S. Environmental Protection Agency, Office of Superfund Remediation and Technology Innovation. January.

## **TABLES**



## Table 2-1 Distribution List



## Upriver Reach Background Investigation Oregon Department of Environmental Quality Willamette River, Oregon

QAPP Recipients	Title	Organization	Email Address
Mark Pugh	DEQ Project Manager	DEQ	mark.pugh@state.or.us
Franziska Landes	Project Technical Lead	DEQ	franziska.landes@state.or.us
Madi Novak	EPA Remedial Project Manager	EPA	novak.elisabeth@epa.gov
Benjamin Leake	EPA Remedial Project Manager	EPA	leake.benjamin@epa.gov
Donald Brown	Quality Assurance Manager	EPA	brown.donaldm@epa.gov
Michael Pickering	Program Manager	MFA	mpickering@maulfoster.com
Josh Elliott	Project Manager	MFA	jelliott@maulfoster.com
Phil Wiescher	Senior Technical Advisor/Project Scientist	MFA	pwiescher@maulfoster.com
Erik Naylor	Project QAM	MFA	enaylor@maulfoster.com
Carolyn Wise	Field Coordinator/On-Site Safety Officer	MFA	cwise@maulfoster.com
Meaghan Pollock	Field Coordinator/On-Site Safety Officer	MFA	mpollock@maulfoster.com
Eric Parker	Sampling Vessel Lead	Research Support Services	eparker@rssincorporated.com
Stephanie Pollen	Project Manager	Bureau Veritas Laboratories	stephanie.pollen@bureauveritas.com
Kent Patton	Project Manager	Apex Laboratories, LLC	kpatton@apex-labs.com
Brian Ford	Project Manager	Pace Analytical Services	brian.ford@pacelabs.com

#### NOTES:

-- = not applicable.

DEQ = Oregon Department of Environmental Quality.

EPA = U.S. Environmental Protection Agency.

MFA = Maul Foster & Alongi, Inc.

QAM = quality assurance manager.

QAPP = quality assurance project plan.

#### Table 2-2 Contact List



## Upriver Reach Background Investigation Oregon Department of Environmental Quality Willamette River, Oregon

Contact Name	Title	Organization	E-mail	Telephone
Mark Pugh	DEQ Site Manager	DEQ	mark.pugh@state.or.us	503-229-5587
Franziska Landes	DEQ Technical Lead	DEQ	franziska.landes@state.or.us	503-229-5538
Madi Novak	EPA Remedial Project Manager	EPA, Region 10	novak.elisabeth@epa.gov	503-326-3277
Benjamin Leake	EPA Remedial Project Manager	EPA, Region 10	leake.benjamin@epa.gov	503-326-2859
Donald M. Brown	EPA Quality Assurance Manager	EPA, Region 10	brown.donaldm@epa.gov	206-553-0717
Michael Pickering	Program Manager	MFA	mpickering@maulfoster.com	(b) (6)
Josh Elliott	Project and Contract Administration Manager	MFA	jelliott@maulfoster.com	(b) (6)
Phil Wiescher	Senior Technical Advisor/Project Scientist	MFA	pwiescher@maulfoster.com	(b) (6)
Carolyn Wise	Field Coordinator/On-Site Safety Officer	MFA	cwise@maulfoster.com	(b) (6)
Meaghan Pollock	Field Coordinator/On-Site Safety Officer	MFA	mpollock@maulfoster.com	(b) (6)
Erik Naylor	Project Quality Assurance Manager	MFA	enaylor@maulfoster.com	(b) (6)
Mary Benzinger	Database Management/Project Chemist	MFA	mbenzinger@maulfoster.com	(b) (6)

#### NOTES:

DEQ = Oregon Department of Environmental Quality.

EPA = U.S. Environmental Protection Agency.

MFA = Maul Foster & Alongi, Inc.



Table 2-3
Analytical Methods, Performance Criteria, and Reporting Limits
Upriver Reach Background Investigation
Oregon DEQ
Willamette River, Oregon

Matrix	Class/ Analysis	CAS	Analyte	Unit	Analytical Method	EDL <sup>(a)</sup>	PQL/LLOQ <sup>(a)</sup>	LCS/LCSD Accuracy <sup>(b)</sup> (%)	LCS/LCSD Precision (RPD)	Laboratory Duplicate Precision (RPD)	Field Duplicate Precision (RPD)	Completeness (%)
Soil/Sediment	PCDD/PCDF	35822-46-9	1,2,3,4,6,7,8-HpCDD	pg/g	EPA 1613B	0.111	0.2	70-140	20	35	50	90
Soil/Sediment	PCDD/PCDF	67562-39-4	1,2,3,4,6,7,8-HpCDF	pg/g	EPA 1613B	0.105	1	78-138	20	35	50	90
Soil/Sediment	PCDD/PCDF	55673-89-7	1,2,3,4,7,8,9-HpCDF	pg/g	EPA 1613B	0.09	1	78-138	20	35	50	90
Soil/Sediment	PCDD/PCDF	39227-28-6	1,2,3,4,7,8-HxCDD	pg/g	EPA 1613B	0.097	1	70-164	20	35	50	90
Soil/Sediment	PCDD/PCDF	70648-26-9	1,2,3,4,7,8-HxCDF	pg/g	EPA 1613B	0.094	1	72-134	20	35	50	90
Soil/Sediment	PCDD/PCDF	57653-85-7	1,2,3,6,7,8-HxCDD	pg/g	EPA 1613B	0.098	1	64-162	20	35	50	90
Soil/Sediment	PCDD/PCDF	57117-44-9	1,2,3,6,7,8-HxCDF	pg/g	EPA 1613B	0.146	1	84-130	20	35	50	90
Soil/Sediment	PCDD/PCDF	19408-74-3	1,2,3,7,8,9-HxCDD	pg/g	EPA 1613B	0.1	1	64-162	20	35	50	90
Soil/Sediment	PCDD/PCDF	72918-21-9	1,2,3,7,8,9-HxCDF	pg/g	EPA 1613B	0.101	1	64-162	20	35	50	90
Soil/Sediment	PCDD/PCDF	40321-76-4	1,2,3,7,8-PeCDD	pg/g	EPA 1613B	0.099	2	70-142	20	35	50	90
Soil/Sediment	PCDD/PCDF	57117-41-6	1,2,3,7,8-PeCDF	pg/g	EPA 1613B	0.092	0.2	80-134	20	35	50	90
Soil/Sediment	PCDD/PCDF	60851-34-5	2,3,4,6,7,8-HxCDF	pg/g	EPA 1613B	0.094	1	70-156	20	35	50	90
Soil/Sediment	PCDD/PCDF	57117-31-4	2,3,4,7,8-PeCDF	pg/g	EPA 1613B	0.087	1	68-160	20	35	50	90
Soil/Sediment	PCDD/PCDF	1746-01-6	2,3,7,8-TCDD	pg/g	EPA 1613B	0.111	1	67-158	20	35	50	90
Soil/Sediment	PCDD/PCDF	51207-31-9	2,3,7,8-TCDF	pg/g	EPA 1613B	0.087	1	75-158	20	35	50	90
Soil/Sediment	PCDD/PCDF	3268-87-9	OCDD	pg/g	EPA 1613B	0.092	1	78-144	20	35	50	90
Soil/Sediment	PCDD/PCDF	39001-02-0	OCDF	pg/g	EPA 1613B	0.108	0.25	63-170	20	35	50	90
Soil/Sediment	Misc	GIS-110-012	Total Organic Carbon	%	EPA 9060		0.1	80-120	20	35	50	90
Soil/Sediment	Misc	Multiple	Grain Size Analysis	%	ASTM-D422 Modified		0.01	NA	20	35	NA	90

0785.01.13, 1/15/2021, Td\_2-3 Analytical Methods, PC, and RLs



Table 2-3
Analytical Methods, Performance Criteria, and Reporting Limits
Upriver Reach Background Investigation
Oregon DEQ
Willamette River, Oregon

NOTES:

-- = not applicable or no value available.

% = percent.

ASTM = ASTM International.

DEQ = Department of Environmental Quality.

EDL = estimated detection limit.

EPA = U.S. Environmental Protection Agency.

HpCDD = heptachlorodibenzo-p-dioxin.

HpCDF = heptachlorodibenzofuran.

HxCDD = hexachlorodibenzo-p-dioxin.

HxCDF = hexachlorodibenzofuran.

LCS = laboratory control sample.

LCSD = laboratory control sample duplicate.

LLOQ = lower limit of quantitation.

MDL = method detection limit.

NA = not applicable.

OCDD = octachlorodibenzo-p-dioxin.

OCDF = octachlorodibenzofuran.

PCDD/PCDF = polychlorinated dibenzodioxins/polychlorinated dibenzofurans.

pg/g = picograms per gram.

PQL/LLOQ = practical quantitation limit/lower level of quantitation.

RPD = relative percent difference.

TCDD = tetrachlorodibenzo-p-dioxin.

TCDF = tetrachlorodibenzofuran.

<sup>(a)</sup> EDLs and PQL/LLOQs for EPA 1613B represent limits provided by the selected laboratory Bureau Veritas Laboraties. Refer to Appendix B of the QAPP for more information about EDLs, PQL/LLOQ and laboratory selection.

(b) Approximate typical limits; percent recovery requirements are laboratory-specific, based on their established control limits.

0785.01.13, 1/15/2021, Td\_2-3 Analytical Methods, PC, and RLs



#### Table 3-1

# PHSS ROD CULs, RALs, and PTW Thresholds for Sediment Upriver Reach Background Investigation Oregon Department of Environmental Quality Willamette River, Oregon

Chemical	Unit	ROD CUL	Site Wide RALs	PTW
Chemicai	Uniii	ROD COL	SHE WIGE RALS	Thresholds
Dioxins/Furans (2,3,7,8-TCDD eq)	pg/g	10	NA	NA
1,2,3,4,7,8-HxCDF	pg/g	0.4	NA	40
1,2,3,7,8-PeCDD	pg/g	0.2	0.8	10
2,3,4,7,8-PeCDF	pg/g	0.3	200	200
2,3,7,8-TCDF	pg/g	0.40658	NA	600
2,3,7,8-TCDD	pg/g	0.2	0.6	10

#### NOTES:

These values were obtained from Tables 17 and 21 of the Portland Harbor Superfund Site ROD, January 2017, and Errata #2 for Portland Harbor Superfund Site Record of Decision ROD Table 17.

CUL = cleanup level.

NA = not applicable.

pg/g = picograms per gram.

PHSS = Portland Harbor Superfund Site.

PTW = principal threat waste.

RAL = remedial action level.

ROD = record of decision.

## Table 4-1 Data Quality Objectives



# Upriver Reach Background Investigation Oregon Department of Environmental Quality Willamette River, Oregon

DQOs	Task	Surface Sediment Sampling
Step 1	State the Problem	The Upriver Reach is used to establish background conditions for the PHSS. There is uncertainty as to whether the background-based ROD riverbank soil/sediment CULs for dioxins/furans developed from previous sediment investigations are representative of background conditions.
Step 2	Identify the Goals of the Study	The new surface sediment data will support an updated evaluation of dioxin/furan background conditions in the Upriver Reach that will inform an evaluation of dioxin/furan soil and sediment cleanup levels established in the ROD.
Step 3	Identify the Information Inputs	Collection of up to 45 three-point composite unbiased surface sediment samples. Bulk samples will be analyzed for dioxins/furans, grain-size distribution, and total organic carbon. Analysis of dioxin/furans and total organic carbon at specific grain size intervals (i.e., No. 10 and 230 sieve).
Step 4	Define the Boundaries of the Study	Geographic Boundary: The samples will be distributed over the Site from RM 16.6 to RM 28.4.  Temporal Boundary: The sediment samples will be collected during tidally-filtered river flow conditions less than 50,000 cubic feet per second.
Step 5	Determine the Analytical Approach	Unbiased background concentrations from samples collected in appropriate and feasible locations will be established by appropriate statistical methods.  The influence of grain size on dioxin/furan concentrations will be evaluated by determining if there is a statistical difference between dioxin/furan and TOC concentrations for the different grain size fractions, evaluating how the relationship between concentrations of dioxin and furan congeners and TOC is impacted by the different sediment grain size fractions, and determining if there is a statistically significant difference in the percent fines between the proposed sampling bulk fraction results and those of the 2018 sampling results (targeted samples with >25 percent fines within the soft sediment polygons).

## Table 4-1 Data Quality Objectives



# Upriver Reach Background Investigation Oregon Department of Environmental Quality Willamette River, Oregon

Step 6	Specify Performance or Acceptance Criteria	The acceptance criteria will entail the attainment of laboratory QA/QC results consistent with Section 4 of the QAPP.
Step 7	Describe the Plan for Obtaining the Data	The surface sediment samples will be collected from three sampling points at each sampling location and homogenized into a three-point composite sample. The rationale for the sampling locations and analytical strategy is further explained in the Work Plan (Section 3) and the FSP (Section 2).

#### NOTES:

CUL = cleanup level.

DQO = data quality objective.

FSP = field sampling plan.

PHSS = Portland Harbor Superfund Site.

QA/QC = quality assurance and quality control.

QAPP = quality assurance project plan.

RM = river mile.

ROD = record of decision.

Site = Upriver Reach of the Willamette River.

TOC = total organic carbon.

#### Table 4-2



# Sample Containers, Methods, Sample Preservation, and Holding Times Upriver Reach Background Investigation Oregon Department of Environmental Quality Willamette River, Oregon

Matrix	Туре	Parameter	Method	Sample Container	Preservation	Archive Preservation (up to 1 year)	Holding Time
	D. III.	Dioxins/Furans	EPA 1613B	1x 8-ounce amber glass jar	Cool to ≤10°C, stored in the dark	Cool to ≤10°C, stored in the dark	1 year from collection to preparation; 1 year from extraction to analysis
	Bulk	Grain Size	ASTM-D422 Modified	1x 16-ounce amber glass jar <sup>(a)</sup>	Do not freeze	NA	None
Sediment		Total Organic Carbon	EPA 9060A	1x 8-ounce amber glass jar	Cool to 0-6°C	NA	28 days from collection to analysis
	Specific Grain Size <sup>(a)</sup>	Dioxins/Furans	EPA 1613B	4x 16-ounce amber glass jars	Cool to ≤10°C, stored in the dark	Cool to ≤10°C, stored in the dark	1 year from collection to preparation; 1 year from extraction to analysis
		Total Organic Carbon	EPA 9060A		Cool to 0-6°C	NA	28 days from collection to analysis
Aqueous		Dioxins/Furans	EPA 1613B	1x 1-liter amber glass	Cool to 0-6°C	Extracts freeze ≤10°C	1 year from collection to preparation; 1 year from extraction to analysis

#### NOTES:

ASTM = ASTM International.

EPA = U.S. Environmental Protection Agency.

NA = not applicable.

<sup>-- =</sup> not applicable.

<sup>°</sup>C = degrees Celsius.

<sup>&</sup>lt;sup>(a)</sup>Additional analyses (dioxin/furans and total organic carbon) will be performed on sediment that passes through a No. 10 and 230 sieve, when sufficient grain size is available for analysis.

# Table 4-3 Field Quality Control Sample Frequency Upriver Reach Background Investigation Oregon Department of Environmental Quality



Analysis	Method	Field Duplicates	MS and/or MS/MSD <sup>(a)</sup>	Equipment Blanks	Temperature Blanks
Dioxin/Furans	EPA 1613B	1 per 20 samples	NA <sup>(b)</sup>	1 per 20 samples	1 per cooler
Grain Size	ASTM-D422 Modified	NA	NA	NA	1 per cooler
Total Organic Carbon	EPA 9060A	1 per 20 samples	NA	NA	1 per cooler

Willamette River, Oregon

NOTES:

ASTM = ASTM International.

DEQ = Oregon Department of Environmental Quality.

EPA = U.S. Environmental Protection Agency.

MS = matrix spike.

MSD = matrix spike duplicate.

NA = not applicable.

<sup>&</sup>lt;sup>(a)</sup>No equipment blanks are required for disposable or dedicated field sampling equipment. Equipment blanks will target about one per week or one per piece of equipment for overall project average of one per 20 samples.

<sup>(</sup>b) MS/MSD are not required when isotope dilution is used.

# Table 4-4 Laboratory Quality Control Sample Frequency Upriver Reach Background Investigation Oregon Department of Environmental Quality Willamette River, Oregon



Analysis	Method	Method Blanks	LCS and/or LCS/LCSD	Laboratory Duplicate	Labeled Standards	Puget Sound Sediment Reference Material
Dioxin/Furans	EPA 1613B	One per extraction batch	One per extraction batch	One per extraction batch or 20 samples	Every sample	Once per project
Grain Size	ASTM D422- Modified	NA	NA	One per extraction batch or 20 samples	NA	NA
Total Organic Carbon	EPA 9060A	One per extraction batch	One per extraction batch	One per extraction batch or 20 samples	NA	NA

NOTES:

ASTM = ASTM International.

EPA = U.S. Environmental Protection Agency.

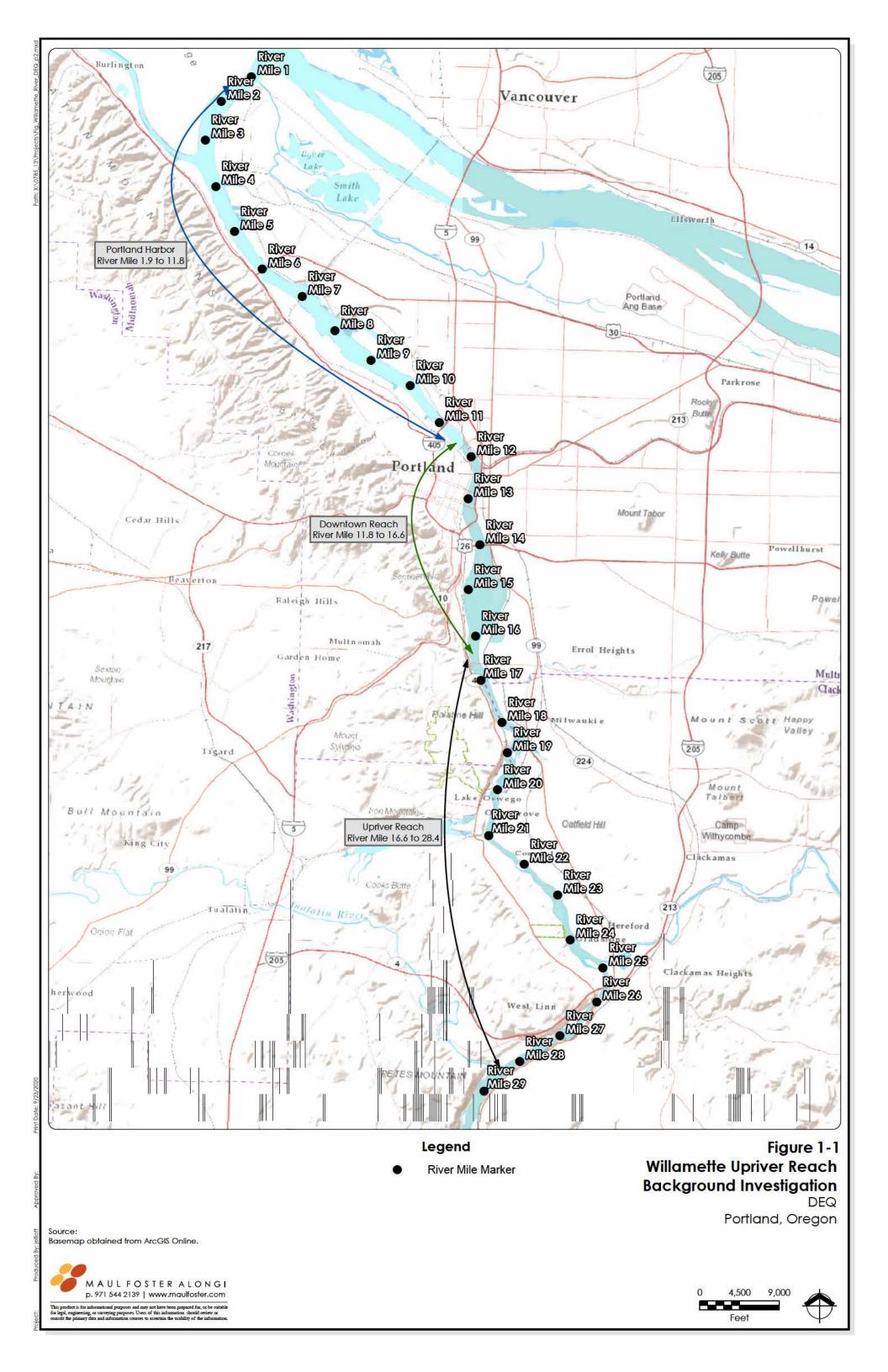
LCS = laboratory control sample.

LCSD = laboratory control sample duplicate.

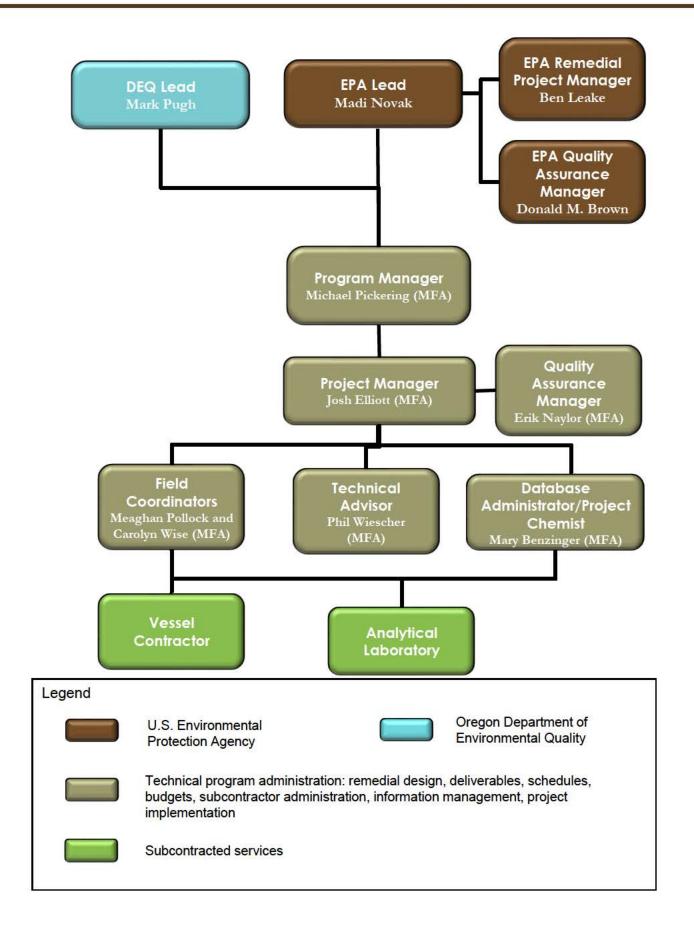
NA = not applicable.

## **FIGURES**





# Figure 2-1 Organization Chart Upriver Reach Background Investigation Oregon Department of Environmental Quality Willamette River, Oregon



## APPENDIX A

SUPPLEMENTAL LABORATORY INFORMATION: LABORATORY METHOD SUMMARIES





#### **Document Information**

Document Number: ENV-SOP-MTJL-0183	Revision: 02
Document Title: Total Organic Carbon In Soils (loss of w	eight on ignit.
Department(s): Wet Chemistry	
Date Information	
Effective Date: 23 Jan 2020	
Notes	
Document Notes:	

All Dates and Times are listed in: Central Time Zone

#### Signature Manifest

Document Number: ENV-SOP-MTJL-0183 Revision: 02

Title: Total Organic Carbon In Soils (loss of weight on ignit.

All dates and times are in Central Time Zone.

#### ENV-SOP-MTJL-0183

#### **QM** Approval

Name/Signature	Title	Date	Meaning/Reason
James Brownfield (006598)	Compliance Director		
Steven Miller (006597)	QA Director	19 Dec 2019, 05:17:25 PM	Approved

#### **Management Approval**

Name/Signature	Title	Date	Meaning/Reason
John Davis (006579)	Manager - Operations	22 Jan 2020, 02:05:15 PM	Approved



TITLE: Total Organic Carbon in Soils by Loss on Ignition

**TEST METHOD:** ASTM F1647 and USDA LOI **ISSUER:** Pace National – Mt. Juliet, Tennessee

COPYRIGHT © Pace Analytical Services, LLC.

#### 1.0 Scope And Application

- 1.1 This procedure is applicable to the determination of Soil Organic Matter (SOM) in non-aqueous liquids including soils, sludges, and sediments. Total Organic Carbon (TOC) can be estimated from the obtained Soil Organic Matter result.
- 1.2 This procedure is applicable to samples with a Total Organic Carbon concentration greater than or equal to 1000 mg/kg or 0.1%.
- 1.3 An estimate of the Total Organic Carbon contained within a given sample is determined by applying the Van Bemmelen Factor, based on the assumption that Organic Matter contains 58% organic carbon. There is no universal conversion factor, as the factor varies from soil to soil; therefore, the TOC result generated by this method is not intended for precise work.

Total Organic Carbon=
$$\frac{\text{Soil Organic Matter}}{1.724}$$

Therefore, using the inverse:

Total Organic Carbon=Soil Organic Matter×0.58

#### 2.0 Summary of Method

2.1 A 50-100g aliquot of solid material is placed in a pre-weighed crucible and weighed. The sample is dried at 105°C ± 5°C and re-weighed. The dried sample or an aliquot is ignited in a muffle furnace at 360°C for USDA projects or 440C for ASTM. The remaining residue following ignition is cooled and re-weighed to constant weight. The weight of sample lost by ignition is calculated and a correction factor is applied to determine the estimated Total Organic Carbon content in mg/kg or percent.

#### 3.0 Interferences

- 3.1 Analytical bias may result from volatilization of substances other than organic materials or from incomplete oxidation of carbonaceous materials.
- 3.2 LOI is strongly dependent on the temperature length of the ignition time.

#### 4.0 Definitions

- 4.1 <u>Total Organic Carbon (TOC)</u> Refers to the amount of organic carbon present within sediment/soil.
- 4.2 <u>Soil Organic Matter</u> The organic fraction of soil, including plant, animal and microbial residues at all stages of decomposition in addition to the relatively resistant soil humus.
- 4.3 Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

#### 5.0 Health and Safety

5.1 The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.



TITLE: Total Organic Carbon in Soils by Loss on Ignition

**TEST METHOD:** ASTM F1647 and USDA LOI **ISSUER:** Pace National – Mt. Juliet, Tennessee

COPYRIGHT © Pace Analytical Services, LLC.

- 5.2 The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (HSE) policies and procedures specified in this SOP and in the Pace National Chemical Hygiene / Safety Manual.
- 5.3 Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.
- 5.4 Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids in a fume hood whenever possible with additional PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.
- 5.5 Contact your supervisor or local HSE coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

#### 6.0 Sample Collection, Preservation, Holding Time, And Storage

- 6.1 Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.
- 6.2 Glass 2oz. or 4oz. jars with Teflon™-lined lids are acceptable.
- 6.3 Samples should be stored at ≤6°C (not frozen). The hold time is 28 days.

#### 7.0 Equipment And Supplies

- 7.1 Crucibles 20 to 100 mL, ignited at 360°C or 440C and stored desiccated
- 7.2 Muffle furnace capable of reaching and maintaining 440°C, Thermolyne 30400 or equivalent
- 7.3 Drying oven VWR™ 1305U, or equivalent
- 7.4 Desiccator Nalgene 5317-0180 or equivalent
- 7.5 Analytical Balance capable of reading 0.1mg accurately, Mettler AG204 or equivalent

#### 8.0 Reagents And Standards

8.1 All reagents and standards must be recorded in the appropriate preparation log and assigned a unique number. See ENV-SOP-MTJL-0041, Standard Logger – Tree Operation. Additional information regarding reagent preparation can be found in the Standards Logger (Tree) digital archive system. All spiking solutions and surrogate standard solutions should be replaced at least every six months or sooner if a problem is detected unless otherwise noted.



TITLE: Total Organic Carbon in Soils by Loss on Ignition

**TEST METHOD:** ASTM F1647 and USDA LOI **ISSUER:** Pace National – Mt. Juliet, Tennessee

COPYRIGHT © Pace Analytical Services, LLC.

8.2 Commercially prepared LCS – ERA Nutrients in Soil, catalog number 542, or equivalent.

Critical Note: Each analyst is responsible for making sure that any newly opened LCS is not used until the certified value has been verified to be correct in PrepData and/or Tree. This step is essential to the process and cannot be forgotten or omitted. If you are unsure about this process, see your supervisor before beginning a workgroup.

#### 9.0 Procedure

- 9.1 Place a 20 or 100mL dried and desiccated crucible on the balance and record the weight.
- 9.2 For each field sample, mix the entire sample well and add at least 10g (50g preferred) to crucible. Record weight of each sample plus crucible.
  - 9.2.1 For the method blank, weigh an empty crucible. Record the weight.
  - 9.2.2 For the LCS, weigh duplicate 10g aliquots of the purchased LCS in separate preweighed crucibles. Record both of these weights.
  - 9.2.3 For sample duplicates, prepare secondary aliquots of field samples at a frequency of 1 per every 10 field samples prepared.
- 9.3 Place the crucibles in the drying oven and dry overnight i.e. at least 16 hrs. at 105°C ± 5°C.
- 9.4 Carefully remove the samples from the oven and cool to room temperature in a desiccator.
- 9.5 Record weight of each dried sample and crucible. If less than 16 hours repeat above until a constant weight is obtained.
- 9.6 Place the crucibles in muffle furnace and heat to a temperature of 360°C or 440°C as needed.
- 9.7 When oven reaches temperature, continue to heat/ignite the samples for at least two hours. If less than 16 hours verify constant weight obtained. Per the experience of the analyst, additional time may be added when high organic matter content is anticipated.
- 9.8 Return the samples to the desiccator for cooling.
- 9.9 Record the final weight of each sample and crucible.
- 9.10 Calculate the SOM or TOC in mg/kg or %, as necessary.

#### 10.0 Data Analysis And Calculations

10.1 The Loss of Weight on Ignition (LOI) is calculated using the following equation:

LOI (mg/kg)=
$$\frac{\text{(A-B)}}{\text{(A-C)}} \times 1,000,000$$

Where:

A = weight of dried sample (105°C) and dish in g

B = weight of ignited sample (360 or 440°C) and dish in g

C = weight of dish in g

10.2 The total organic carbon content (TOC) is calculated as follows:



TITLE: Total Organic Carbon in Soils by Loss on Ignition

**TEST METHOD:** ASTM F1647 and USDA LOI **ISSUER:** Pace National – Mt. Juliet, Tennessee

COPYRIGHT © Pace Analytical Services, LLC.

Total Organic Carbon (mg/kg)=LOI (mg/kg)×0.58

10.3 See the Laboratory Quality Assurance Manual for equations for common calculations.

#### 11.0 Quality Control and Method Performance

- 11.1 Batches are defined as sets of 1 20 samples. Batch analysis must include the following: 1 method blank, 1 Laboratory Control Sample (LCS), and 1 Sample Duplicate for every 10 samples.
- 11.2 Blanks The concentration of the blank must be less than the method detection limit.
- 11.3 <u>LCS</u> The % recovery must be within the control limits on the COA from the vendor that produced this reference material and the relative % difference (RPD) must be ≤20%.
- 11.4 Sample duplicate The relative % difference (RPD) must be ≤20%.
- 11.5 Analyst Qualifications and Training
  - 11.5.1 Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to laboratory ENV-SOP-MTJL-0015, Technical Training and Personnel Qualifications for Chemistry for more information.

#### 12.0 Data Review And Corrective Action

12.1 <u>Blank</u> – A failing blank may be reanalyzed one time. If it still fails, the reason for the failure must be identified and corrected. The entire workgroup must be re-prepped and re-analyzed.

General guidelines for qualifying sample results with regard to method blank quality are as follows:

- If the method blank concentration is less than the MDL and sample results are greater than the RL, then no qualification is required.
- No qualification is necessary when an analyte is detected in the method blank but not in the associated samples.
- If the concentration in a sample is more than ten times the concentration in the method blank, then no qualification is required.
- If the method blank concentration is greater than the MDL but less than the RL and sample results are greater than the MDL, then qualify associated sample results to indicate that analyte was detected in the method blank.
- If the method blank concentration is greater than the RL, further corrective action and qualification is required. An analyst should consult their supervisor for further instruction.
- 12.2 <u>LCS</u> A failing LCS may be re-analyzed once. If the failure persists, the cause of the failure must be identified and corrected and the entire workgroup must be re-prepped and reanalyzed.



TITLE: Total Organic Carbon in Soils by Loss on Ignition

**TEST METHOD:** ASTM F1647 and USDA LOI **ISSUER:** Pace National – Mt. Juliet, Tennessee

COPYRIGHT © Pace Analytical Services, LLC.

12.3 <u>Sample Duplicate</u> – If the RPD is >20%, the sample may be reported with a J3 qualifier. If the RPD is beyond acceptance limits and the sample concentration is <5X the RL, then the value can be flagged with a "P1" qualifier indicating that the RPD calculation is not applicable at that concentration.

#### 12.4 Data Review

- 12.4.1 Pace National's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.
- 12.4.2 The review steps and checks that occur as employee's complete tasks and review their own work is called primary review.
- 12.4.3 All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance with the Pace National's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.
- 12.4.4 A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.
- 12.4.5 Refer to ENV-SOP-MTJL-0014, *Data Handling and Reporting* and ENV-SOP-MTJL-0038, *Data Review* for specific instructions and requirements for each step of the data review process.

#### 12.5 Corrective Action

12.5.1 Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

#### 13.0 Pollution Prevention And Waste Management

- 13.1 Pace National proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.
- 13.2 The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace National's Chemical Hygiene Plan / Safety Manual.



TITLE: Total Organic Carbon in Soils by Loss on Ignition

**TEST METHOD:** ASTM F1647 and USDA LOI **ISSUER:** Pace National – Mt. Juliet, Tennessee

COPYRIGHT © Pace Analytical Services, LLC.

#### 14.0 Modifications

14.1 Not applicable to this SOP

#### 15.0 Responsibilities

- 15.1 Pace National employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace National's policy for temporary departure.
- 15.2 Pace National supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

#### 16.0 Attachments

16.1 Not applicable to this SOP

#### 17.0 References

- 17.1 Methods for the Determination of Total Organic Carbon (TOC) in Soils and Sediments, Brian A. Schumacher, USEPA, April 2002.
- 17.2 USDA Soil Survey Staff. Mineral Content, Method Code 5A (USDA LOI). Kellogg Soil Survey Laboratory Methods Manual, Soil Survey Investigations Report Number 42 (Version 5.0), 495-497, 2014.
- 17.3 USDA Soil Survey Staff. Dry Combustion, Method Code 4H2. Kellogg Soil Survey Laboratory Methods Manual, Soil Survey Investigations Report Number 42 (Version 5.0), 464-465, 2014.
- 17.4 ASTM D2974-07A, Standard Test Methods for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils.



TITLE: Total Organic Carbon in Soils by Loss on Ignition

TEST METHOD: ASTM F1647 and USDA LOI ISSUER: Pace National – Mt. Juliet, Tennessee

COPYRIGHT © Pace Analytical Services, LLC.

#### 18.0 Revision History

#### **Current Version (Pace National):**

Date	Description of Revisions		
12/8/2019	Revised sections 1.1, 1.2, 1.3, 2.1, 3.2, 7.1, 7.2, 9.1, 9.2, 9.2.2, 9.3, 9.5, 9.6, 9.7, 9.10 and 10.1. Deleted sections 3.3, 4.2, 7.5, 17.1 and renumbered as necessary.		

#### Superseded Versions (ESC Lab Sciences SOP #340356C):

Version	Date	Description of Revisions		
0	2/6/04	Origination		
1	7/2/07	Technical and Quality Review		
2	8/17/09	Technical and Quality Review and update; Revised sections 11.3 through 11.6, 12.1; Added sections 7.1, 7.3, 8.6, 9.4, 9.5, 10.4 through 10.6, 11.2, 14.4.		
3	2/23/11	Technical and Quality Review and update; Revised sections 2.1, 5.3, 5.5, 6.2, 7.3, 8.6 through 8.8, 9.4, 9.5, 10.2, and 12.1.		
4	4/6/12	Technical and Quality Review and update; Revised sections 6.1, 6.3, 6.4, 11.1; Added section 6.6.		
5	9/21/12	Technical and Quality Review and update; Revised sections 2.1, 7.3, 8.2, 8.3, 8.4, 8.8, 10.1, and 11.1; Removed section 1.5; Added sections 13.2 and 14.3		
8/21/14		Reviewed with no changes per C. Pfalmer/D. Marlin		
6	12/9/2015	Technical and quality review and update. Header and signature block re-formatting. Revised Sections 7.1, 10.4, 11.3, and 12.2.		
7	6/5/2017	Technical and quality review and update. Header and signature block re-formatting. Revised Sections 1.3, 2.1, 2.2, 2.3, 2.4, 2.5, 3.2, 4.3, 5.3, 5.4, 6.2, 6.4, 6.5, 6.6, 8.1, 8.2, 8.2.1, 8.2.2, 8.3, 8.5, 8.6, 8.7, 8.8, 8.9, 8.10, 9.2, 9.4, 10.1, 10.5, 10.6, 11.2.4, 12.1, 13.1, 13.2, 14.1, and 14.2. Deleted Sections 1.4, 2.6, 2.7, 5.5, 7.3, 8.11, 9.5, 11.6, and 11.6.1.		

#### Superseded Versions (Pace National):

Version	Date	Description of Revisions		
12/14/18	Revised section 8.6, 8.7, 9.1, 1	Technical and quality review and update. Deleted header, footer and signature bar. Revised sections 1.1, 2.1, 2.4, 4.2, 5.4, 6.1, 6.2, 6.3, 6.4, 6.5, 6.6, 7.1, 7.2, 8.2.1, 8.3, 8.6, 8.7, 9.1, 10.1, 10.2, 11.2.4, 11.5, 12.1, 12.2, 13.0, 14.1, 14.2, 14.3, 14.4 and 14.5. Deleted sections 5.3, 13.1 and 13.2. Revised Attachment I.		
6/13/19	Converted SOP over to new format			
8/4/2019	Converted SOP over to new format. Revised section 8.2, 11.3 and 11.4.			

#### APEX LABORATORIES, LLC

### STANDARD OPERATING PROCEDURE APPROVAL SIGNATURE PAGE

Document Title: Grain-Size Analysis of Soils (Full Grain-Size method with hydrometer readings)

Document #: **G - 101** Revision #: **R - 03** Effective Date: **09/27/19**Method / TNI code: ASTM 422-63 (07) / TNI 30030854

Last Review Date:	DO NOT USE AFTER: 09/27/2	09/27/21
Experience of the Control of the Con	(undate +2vr after review/ revision)	

Approval Signatures:

James Johnson Da Subject Review

Jason Woodcock

Date

Date

Technical Review

Kent Patton QA Review

James Johnson Technical Review

Kent Patton QA Review

#### APEX LABORATORIES, LLC

## STANDARD OPERATING PROCEDURE APPROVAL SIGNATURE PAGE

lethodology (RSM)
Effective Date: 10/17/18
DO NOT USE AFTER: 10/17//20 (update +2yr after review/ revision)
10/10/2018



# METHOD SUMMARY

DETERMINATION OF POLYCHLORINATED DIBENZO-P-DIOXINS (PCDDs) AND POLYCHLORINATED DIBENZOFURANS (PCDFs) IN WATER, SOIL, FOOD and BIOTA SAMPLES BY ISOTOPE DILUTION HRGC/HRMS

**BRL SOP-00410** 

Reference Method:

- Based on EPA 1613B



#### **Principle of Method**

The PCDDs/DFs are extracted from solid samples with toluene and from aqueous samples with methylene chloride. Following extraction, the samples are cleaned up and passed through a series of columns that remove, by reaction and/or selective adsorption, the bulk of the organic matrix coextracted with the PCDDs and PCDFs. 37Cl4 2,3,7,8 TCDD is added to each extract to measure the efficiency of the cleanup process. Sample cleanups may include back extraction with acid and/or base, and gel permeation, alumina, silica gel, Florisil and activated carbon chromatography.

The resulting fraction is concentrated to a known volume for analysis. Qualitative/quantitative analysis for PCDDs and PCDFs is performed using separation by High Resolution Capillary Gas Chromatography (HRGC) and measured by High Resolution Mass Spectrometry (HRMS). PCDDs/DFs are identified by comparing GC retention times and the ion abundance ratios of the m/z's with the corresponding values obtained for authentic standards.

The analyte concentration is determined using HRGC/HRMS and an isotope dilution technique. Quantitation is based on the use of carbon 13 labelled internal standards and relative response factors (RRFs). Total PCDDs and PCDFs are reported as the sum of the individual isomers, corrected for internal standard recoveries. The toxic equivalence (TEQs) for the sample is the sum of the individual 2,3,7,8 isomers found multiplied by its specific Toxic Equivalence Factor (TEF).

Sample Handling and Hold-Time for Extraction and Analysis

Matrix	Container	Minimum Volume	Time to Extract	Time to Analysis
Waters	4 x 1L amber glass bottle	800mL	30 days	45 days
Solids/Sludge	100mL amber glass	2 x 5 grams	30 days	45 days
Tissue	100mL amber glass	2 x 20 grams	30 days	45 days

### **Q JALITY SYSTEMS MANUAL**

for

## **Apex Laboratories, LLC**

6700 SW Sandb irg St Tigard, Oregon 97223 503-718-2323

SO <sup>o</sup> Number: Q-001

Re ision: 8

Effective Date: April 1, 2020

Name	Title	Signatures	Date	
Philip Nerenberg	Laboratory Director	Philippentry	3/31/20	
Estella Rieben	Quality Systems Manager	Etella K. Rieben	3/31/20	
, David Jack	Technical/Operations Manager	DA	4/2/20	
Kent Patton	Director of Technical Services	KINM	3/31/20	

#### **Table of Contents**

Section	Title	Page	Effective Date		
	TITLE PAGE 1 04/01/2020				
	No. of the state o	2			
	TABLE OF CONTENTS		04/01/2020		
1.0	INTRODUCTION, SCOPE, AND APPLICABILITY				
***	1.1 Introduction	3	06/02/2018		
	1.2 Scope	3	06/02/2018		
2.0	NORMATIVE REFERENCES	- 1			
	2.1 Table of Contents, References, and Appendices	3	04/01/2020		
3.0	TERMS AND DEFINITIONS	*			
	3.1 Terms and Definitions	4	06/02/2018		
	3.2 Sources	6	06/02/2018		
4.0	MANAGEMENT REQUIREMENTS				
	4.1 Organization	7	02/11/2019		
	4.2 Management	10	04/01/2020		
	4.3 Document Control	18	06/02/2018		
	4.4 Review of Request, Tenders, and Contracts	20	06/02/2018		
	4.5 Subcontracting of Environmental Tests	22	06/02/2018		
	4.6 Purchasing Services and Supplies	23	06/02/2018		
	4.7 Service to the Client	24	06/02/2018		
	4.8 Complaints	24	06/02/2018		
	4.9 Control of Nonconforming Environmental Testing Work	25	06/02/2018		
	4.10 Improvement	29	06/02/2018		
	4.11 Corrective Action	29	06/02/2018		
	4.12 Preventive Action	30	06/02/2018		
	4.13 Control of Records	30	04/01/2020		
	4.14 Internal Audits	34	06/02/2018		
	4.15 Management Reviews	36	06/02/2018		
	4.16 Data Integrity Investigations	36	06/02/2018		
5.0	TECHNICAL REQUIREMENTS				
	5.1 General	37	06/02/2018		
	5.2 Personnel	37	06/02/2018		
	5.3 Accommodation and Environmental Conditions	39	04/01/2020		
	5.4 Environmental Methods and Method Validation	40	06/02/2018		
	5.5 Calibration Requirements	42	06/02/2018		
	5.6 Measurement Traceability	48	06/02/2018		
	5.7 Collection of Samples	49	06/02/2018		
	5.8 Handling of Samples and Test Items	50	06/02/2018		
	5.9 Quality Assurance for Environmental Testing	50	06/02/2018		
	5.10 Reporting the Results	60	06/02/2018		
	APPENDICES				
	A Glossary	61	06/02/2018		
	B Organization Chart	67	02/11/2019		
	C Apex Laboratories, LLC Floor Plan	68	02/11/2019		
	D Example Chain of Custody	70	02/11/2019		
	E Example Cooler Receipt Form	71	02/11/2019		
	F Certifications and Scopes of Accreditation	72	04/01/2020		
	G Changes from Previous Revision	150	04/01/2020		
		,			

## APPENDIX B

LABORATORY SURVEY RESULTS MEMORANDUM





To: Mark Pugh and Danielle Johnson, DEQ Date: December 16, 2020

From: Josh Elliott and Erik Naylor, MFA Project No.: 0785.13.01

RE: Final Laboratory Survey Results—Task Order No. 73-18-15-001 Willamette Upriver Reach

Background Investigation

Maul Foster & Alongi, Inc. (MFA) prepared this Laboratory Survey Results (LSR) memorandum on behalf of the Oregon Department of Environmental Quality (DEQ) to support the Willamette Upriver Reach Background Investigation. This investigation will be conducted in the Willamette River from river miles 16.6 to 28.4, from approximately the Sellwood Bridge in Portland, Oregon, to the confluence of the Tualatin and Willamette rivers in West Linn, Oregon (Upriver Reach) (Figure 1).

This LSR memo describes the results of the laboratory survey that MFA conducted as detailed in the Laboratory Survey Approach and Methods memorandum issued to DEQ (MFA, 2020). As described in the memorandum, there are four components of the evaluation, which are summarized herein:

- Limit Solicitation and Laboratory Interview
- Data Review
- Validation Review
- Limit Determination

A helpful set of definitions and results is provided below.

#### **DEFINITIONS**

Critical to understanding the LSR is consistent use of terminology. Relevant terms are defined below.

• Method detection limit (MDL)—The MDL is defined as the minimum measured concentration of a substance that can be reported with 99 percent confidence that the measured concentration is distinguishable from method blank results (U.S. Environmental Protection Agency [EPA], 2016c). MDLs are required for Clean Water Act methods (Method 1613B) but no longer required for EPA Method SW-846 analyses (Method 8290A); irrespective of requirements, MDL studies are still often conducted by laboratories using SW-846 methods.

- Estimated detection limit (EDL)—The sample- and analyte-specific EDL is a laboratory's estimate of the concentration of a given analyte that would have to be present to produce a signal with a peak height of at least 3 times the background noise signal level (EPA, 2016b).
- Reporting detection limit (RDL)—For the purposes of this memorandum, RDL includes both the MDL and the EDL.
- Practical quantitation limit (PQL)/lower limit of quantitation (LLOQ)—The lowest concentration that can be reliably measured within specified limits of precision, accuracy, representativeness, completeness, and comparability during routine laboratory operating conditions. The PQL is usually the lowest concentration used to calibrate an instrument after being adjusted for sample volume, sample extract volume, extract cleanup, and injection volume. PQLs are often three to ten times the MDL. Under SW-846 Final Update V (EPA, 2020), a PQL may be considered equivalent to the LLOQ except that the LLOQ is the lowest concentration used to calibrate the instrument. Minimum level (ML) is also a term that may be used instead of LLOQ.
- Method reporting limit (MRL)-For the purposes of this memorandum, MRL includes PQL/LLOQ and ML.
- Estimated Maximum Potential Concentration (EMPC)—An EMPC is a value calculated for a reported analyte when the signal-to-noise ratio is at least 2.5:1 for both quantitation ions, but the ion abundance ratio criteria used for analyte confirmation are not met, or when polychlorinated diphenyl ether interference has occurred (EPA, 2016b). An EMPC value represents the maximum possible result of an analyte that could not be positively identified or a result that co-eluted with diphenyl ethers. The inability to positively identify the analyte could be a result of matrix interference, a coeluting compound, or low response.

#### LIMIT SOLICITATION AND LABORATORY INTERVIEWS

For this evaluation, MFA contacted the following laboratories:

- Vista Analytical Laboratory in El Dorado Hills, California
- Pace Analytical Services' Dioxin Laboratory in Minneapolis, Minnesota
- Eurofins Test America in Knoxville, Tennessee and Sacramento, California
- Bureau Veritas Laboratories in Calgary, Alberta, Canada
- Alpha Analytical in Westborough, Massachusetts
- ALS Global's Laboratory in Houston, Texas
- SGS Axys in Sidney, British Columbia, Canada
- Pacific Rim Laboratories in Surrey, British Columbia, Canada
- Cape Fear Analytical in Wilmington, North Carolina

- Ceres Analytical Laboratory, Inc., in El Dorado Hills, California
- Analytical Resources, Incorporated, in Tukwila, Washington

For each laboratory, MFA requested the following information:

- Available dioxin/furan reporting limits, including EDLs, MDLs, and PQLs/LLOQs. A summary of this information is provided in Table 1.
- Available standard and expedited turnaround times.
- Pricing for dioxin/furan analysis.
- Example level 2 and level 4 data packages.
- Example electronic data deliverables (EDDs).
- Accreditations.

Reporting limits are summarized in Table 1. The remaining information is summarized in Table 2.

MFA also interviewed a dioxin/furan expert from each laboratory to discuss the following:

- A detailed explanation of the specific analytical method for analysis of dioxin/furans
- Laboratory recommendations and procedures associated with achieving the lowest reporting limits possible for this project
- Current analytical limits and deviations from those limits over time
- Common challenges associated with analysis of dioxins/furans, specifically those that affect reporting limits, and any procedures used to overcome challenges (e.g., extract cleanup)
- History of analyzing samples associated with the Portland Harbor Superfund Site

Laboratory interviews provided MFA a chance to discuss the project and qualitatively gauge each laboratory's dioxin/furan capabilities and the level to which the laboratory may engage during the project. A summary of interview discussions is provided in Table 3.

Each laboratory listed above, with the exception of two, responded to MFA's information request (responding laboratories) and was interviewed. The exceptions were Alpha Analytical, which did not respond to MFA's request for information, and Ceres Analytical Laboratory, Inc., which responded but did not attend the scheduled interview. Responding laboratories each confirmed their capability to use analytical methods 1613B or 8290A (with a preference for 1613B for quality control purposes and best reporting limits), meet the requested reporting requirements (including preparation of level 2 and level 4 data packages), and generate EDDs according to the format provided by MFA. In

addition, each of the responding laboratories holds some type of accreditation. Many laboratories are accredited by the Oregon Environmental Laboratory Accreditation Program, while others have accreditation from one or more of the following programs:

- National Environmental Laboratory Accreditation Program (accreditation from states other than Oregon)
- Department of Defense Environmental Laboratory Accreditation Program
- International Organization for Standardization 17025
- Washington State Department of Ecology
- Canadian Association of Laboratory Accreditation

Many laboratories hold all these accreditations. A summary of laboratory accreditations is provided in Table 2.

The pricing information provided by responding laboratories was for typical dioxin/furan analysis only and did not account for additional costs associated with level 4 reporting packages, shipping of samples, and expedited turnaround times. Basic and expedited turnaround times were consistent for all responding laboratories (three to four weeks for standard, one week expedited). The laboratories acknowledge that expedited turnaround time availability depends on laboratory capacity at the time of the project and that the associated price is variable. Prices provided by laboratories during this survey are likely to differ from prices that come from a bid solicitation.

#### **DATA REVIEW**

MFA reviewed several recent and available datasets to determine whether the dioxin/furan limits had been achieved in previous analyses of Willamette River sediments. MFA identified five such datasets where sediments had been analyzed for dioxins/furans:

- Remedial Investigation/Feasibility Study (RI/FS) sample data from the Final Portland Harbor RI/FS—Remedial Investigation Report (EPA, 2016a)
- 2017 DEQ Upriver sample date from the Final Field and Data Report—Upriver Reach Sediment Characterization (GSI and Hart Crowser, 2018)
- 2018 Baseline Pre-RD Group from the Pre-Remedial Design Footprint Report (AECOM and Geosyntec, 2019)
- 2018 Baseline EPA Split sampling database
- 2018 DEQ orphan data from the Field and Data Report—Upriver Reach Sediment Investigation (Hart Crowser, 2020)

Mark Pugh and Danielle Johnson, DEQ December 16, 2020 Page 5

MFA located dioxin/furan results for 2,015 samples in the identified data sets and compiled the associated results, reporting limits, river miles, collection date, laboratory, validator, qualifiers, and other relevant metadata. The findings of the data review are summarized in Table 4. The data were used to conduct the aspects of the limit determination, as discussed later in this memo. The findings of the data review were also used to inform the validation review, as described below.

#### **VALIDATION REVIEW**

MFA reviewed a random selection of available laboratory reports, including those that were referenced in validation reports of interest; validation reports; and validation qualifiers associated with the data sets described in the previous section. These data were evaluated to identify whether limits were elevated, and if so if this resulted from method blank contamination, EMPCs, or other data quality issues that could potentially elevate EDLs, MDLs, and PQLs above the laboratory proposed limits. A summary of the findings for each data set is included in Table 5.

#### LIMIT DETERMINATION

MFA compared the limits for each dioxin/furan congener provided by responding laboratories against the findings of the data and validation reviews to determine the difference between laboratory proposed limits and limits observed in previously analyzed Willamette River sediment samples. Specifically, MFA compared:

- Limits from each responding laboratory (Table 1)
- Limits from laboratories included in the data and validation review against the limits solicited from the same laboratory (Table 6)
- Limits between and among all laboratories included in the data and validation review (Table 6)
- Variability in limits between congeners (Figures 2 through 4), using 2,3,7,8-TCDD; 2,3,7,8-TCDF; 1,2,3,7,8-PeCDD; 2,3,4,7,8-PeCDF; and 1,2,3,4,7,8-HxCDF as examples.

#### Laboratory Solicitation Limits

Table 1 provides a summary of the limits provided by responding laboratories. TCDD and TCDF congeners generally have the lowest limits, followed by penta, hepta, and hexa congeners, and lastly octa congeners. The laboratory provided EDLs and MDLs sometimes vary by an order of magnitude between laboratories. Laboratories acknowledged that RDLs have not significantly changed in the past ten years.

EDLs provided by laboratories are based on their theoretical lowest limit (i.e., what they generally report for sediment matrices or averages from the past year of sediment analyses). Therefore, EDLs could be skewed, as some laboratories are likely to see more (or fewer) impacted samples than others. During the interviews, laboratories were confident that their EDLs are realistically achievable but

Mark Pugh and Danielle Johnson, DEQ December 16, 2020 Page 6

acknowledged that variability can occur based on sample matrix, moisture content, and presence of interferences.

A comparison of MDLs provides information about how well the laboratories perform in a controlled environment and can be helpful in determining laboratory and analyst proficiency. However, MDLs are not an accurate predictor of the laboratory's ability to handle sediment matrices with high concentrations of dioxins/furans, interferences, or high moisture contents.

The PQLs/LLOQ provided by the laboratories are less likely to be impacted by sample interferences when compared to the EDLs and MDLs, but this could change should less sample volume than the standard extraction volume is extracted. The laboratories would be expected to extract less sample volume in cases where matrix interferences were very high, or dioxin/furan concentrations were above the high end of the calibration. Laboratory PQL/LLOQs ranged significantly, as some laboratories include calibration points below the 1613B method requirements.

### Data Review Limits

MFA reviewed the datasets described above and filtered for non-detect results (identified with a U qualifier) to identify all RDLs reported for the datasets. Table 6 summarizes various statistics for RDLs obtained, as well as associated PHSS Record of Decision (ROD) (EPA, 2017) cleanup levels (CULs); principal threat waste (PTW) thresholds; remedial action levels (RAL); and the detection limits solicited from the laboratories. Table 6 also provides the laboratory that generated the data associated with each study (when known) and those limits are compared to the responding laboratories limits. The RDLs from the Upriver Reach and the PHSS were compared for the five dioxin congeners with associated ROD criteria (see Figure 2). The results indicate RDLs tend to be higher in the PHSS which may reflect more matrix interferences in PHSS sediments due to widespread presence of contaminants. Figure 3 shows how RDLs differ between datasets reviewed and that the RDL results are typically below CULs established in the ROD. It is noted that EMPCs were not handled consistently between data sets and EMPCs validated as "I" in one data set and "U" in another data set result in a potential bias when comparing datasets. The elevated RDLs for the 2018 Baseline EPA Split event are likely due to a lower than usual amount of sample volume extracted due to interferences. In addition, MDLs were compiled when sufficient documentation was available. The MDLs reported were equivalent to the RDLs in some cases, and in some cases the MDLs are suspected to be EDLs. In other cases, MDLs were not available. A comparison of RDLs and MDLs for the Upriver Reach is shown in Figure 4. MDLs as well as RDLs for the Upriver Reach are typically below CULs established in the ROD.

### CONCLUSIONS

When solicited laboratory limits are compared to dioxin/furan congener-specific PHSS ROD action levels (including PHSS ROD (EPA, 2017) CULs, PTW thresholds, and RALs), the RALs are achievable when compared to laboratory-provided EDLs and MDLs (assuming that matrix interferences do not elevate them significantly) but not when compared to PQLs/LLOQs. There are

Mark Pugh and Danielle Johnson, DEQ December 16, 2020 Page 7

exceptions, however, as documented in Table 1. PHSS PTW thresholds are achievable when compared to laboratory EDLs, MDLs, and PQLs/LLOQs.

Congener-specific RDLs reviewed from existing data sets indicate that PTW thresholds have been achieved historically and that CULs and RALs have been achieved much of the time in the reviewed datasets, with the exception of the 2018 Baseline EPA Split event, which had elevated RDLs due to a lower than usual amount of sample volume extracted because of interferences. Many of those data are likely to be J qualified for detections between the EDL or MDL and the PQL/LLOQ or for some other reason that occurred during validation. It should be noted that the qualification method used for EMPCs (U or J) could potentially bias RDLs, depending on the number of EMPC results that were qualified compared to the overall result count.

Some laboratories can meet PHSS CULs and RALs, with some generated data qualified J as estimated between the EDL/MDL and the PQL/LLOQ. Most laboratories can achieve the PHSS PTW threshold, with their PQL/LLOQ resulting in data not qualified J as estimated.

### REFERENCES

AECOM and Geosyntec. 2019. Revised pre-remedial design footprint report—Portland Harbor pre-remedial design investigation and baseline sampling. Prepared for U.S. Environmental Protection Agency by AECOM, Portland, Oregon, and Geosyntec, Seattle, Washington. <a href="http://ph-public-data.com/document/PHRD">http://ph-public-data.com/document/PHRD</a> 2019. April 8.

EPA. 2016a. Final Portland Harbor RI/FS—remedial investigation report. U.S. Environmental Protection Agency, Seattle, Washington. <a href="http://ph-public-data.com/document/CDMSmith2018">http://ph-public-data.com/document/CDMSmith2018</a>. February 8.

EPA. 2016b. EPA Superfund contract laboratory program, national functional guidelines for high resolution Superfund methods data review. EPA 542-B-16-001. U.S. Environmental Protection Agency, Office of Superfund Remediation and Technology Innovation. April.

EPA. 2016c. Definition and procedure for the determination of the method detection limit. Rev. 2. U.S. Environmental Protection Agency, Office of Water. December.

EPA. 2017. Record of Decision—Portland Harbor Superfund Site. U.S. Environmental Protection Agency Region 10, Seattle, Washington. January.

EPA. 2020. The SW-846 compendium. U.S. Environmental Protection Agency. Washington, DC. <a href="https://www.epa.gov/hw-sw846/sw-846-compendium">https://www.epa.gov/hw-sw846/sw-846-compendium</a> (accessed August 31, 2020). July 31.

GSI and Hart Crowser. 2018. Final field and data report—upriver reach sediment characterization. Prepared for Oregon Department of Environmental Quality by GSI Water Solutions, Portland,

Project No. 0785.13.01

Oregon, and Hart Crowser, Portland, Oregon. <a href="http://ph-public-data.com/document/GSI">http://ph-public-data.com/document/GSI</a> 2018. May 8.

Hart Crowser. 2020. Field and data report—upriver reach sediment investigation. Prepared for Oregon Department of Environmental Quality by Hart Crowser, Portland, Oregon. <a href="http://ph-public-data.com/document/DEQ2019">http://ph-public-data.com/document/DEQ2019</a>. January 31.

MFA. 2020. Memorandum re: laboratory survey approach and methods—Task Oder No. 73-18-15-001 Willamette River upriver reach background investigation. Prepared for the Oregon Department of Environmental Quality by Maul Foster & Alongi, Inc., Portland, Oregon. September 24.

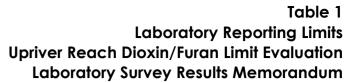
## **TABLES**







Laboratory:	PH	ROD Limits (po	g/g)	Pacif	ic Rim Lab	oratories		SGS Axy	'S	Burea	u Veritas La	boratories	Vista .	Analytical L	aboratory.	Analy	tical Resou	rces, Inc.
Reporting Limit (pg/g):	CUL <sup>(1)</sup>	PTW <sup>(2)</sup>	RALs <sup>(2)</sup>	EDL	MDL	PQL/LLOQ	EDL	MDL	PQL/LLOQ	EDL	MDL	PQL/LLOQ	EDL	MDL	PQL/LLOQ	EDL	MDL	PQL/LLOQ
2,3,7,8-TCDD	0.2	10	0.6	0.043	0.01	0.2	0.05	0.25	0.2	0.111	0.181	0.2	0.20	0.269	0.5		0.14	1
1,2,3,7,8-PeCDD	0.2	10	0.8	0.013	0.05	1	0.05	0.53	1	0.105	0.242	1	0.23	0.729	2.5		0.18	1
1,2,3,4,7,8-HxCDD				0.008	0.09	1	0.05	0.69	1	0.09	0.211	1	0.30	0.654	2.5		0.18	1
1,2,3,6,7,8-HxCDD				0.006	0.1	1	0.05	0.49	1	0.097	0.228	1	0.39	0.593	2.5		0.15	1
1,2,3,7,8,9-HxCDD				0.007	0.06	1	0.05	0.6	1	0.094	0.203	1	0.39	0.619	2.5		0.22	1
1,2,3,4,6,7,8-HpCDD				0.013	0.09	1	0.05	0.86	1	0.098	0.177	1	0.43	0.615	2.5		0.56	2.5
OCDD				0.016	0.2	1	0.05	3.47	2.0	0.146	0.185	1	1.63	1.186	5	-	4.3	10
2,3,7,8-TCDF	0.406	600		0.029	0.01	0.2	0.05	0.22	0.2	0.1	0.194	1	0.15	0.168	0.5		0.063	1
1,2,3,7,8-PeCDF				0.014	0.07	1	0.05	0.56	1	0.101	0.209	1	0.21	0.729	2.5		0.15	1
2,3,4,7,8-PeCDF	0.3	200	200	0.011	0.07	1	0.05	0.55	1	0.099	0.278	2	0.23	0.840	2.5	-	0.15	1
1,2,3,4,7,8-HxCDF	0.4	40		0.005	0.09	1	0.05	0.49	1	0.092	0.227	0.2	0.25	0.528	2.5	-	0.14	1
1,2,3,6,7,8-HxCDF				0.006	0.04	1	0.05	0.53	1	0.094	0.258	1	0.26	0.721	2.5		0.18	1
1,2,3,7,8,9-HxCDF				0.005	0.08	1	0.05	0.52	1	0.087	0.204	1	0.28	0.707	2.5		0.21	1
2,3,4,6,7,8-HxCDF				0.01	0.07	1	0.05	0.53	1	0.111	0.172	1	0.33	0.666	2.5	-	0.11	1
1,2,3,4,6,7,8-HpCDF				0.005	0.05	1	0.05	1.06	1	0.087	0.179	1	0.38	0.678	2.5	-	0.21	1
1,2,3,4,7,8,9-HpCDF				0.006	0.03	1	0.05	0.51	1	0.092	0.244	1	0.32	0.548	2.5	-	0.16	1
OCDF				0.009	0.17	2	0.05	1.18	2.0	0.108	0.743	0.25	0.62	4.507	5		1.1	2.5





Laboratory:	PH	ROD Limits (p	g/g)	Car	oe Fear Ana	lytical	Pace	Analytical S	Services	Euro	ofins/Test An	nerica		ALS Globa	I	Ceres	Analytical L	aboratory
Reporting Limit (pg/g):	CUL <sup>(1)</sup>	PTW <sup>(2)</sup>	RALs <sup>(2)</sup>	EDL	MDL	PQL/LLOQ	EDL	MDL	PQL/LLOQ	EDL	MDL	PQL/LLOQ	EDL	MDL	PQL/LLOQ	EDL	MDL	PQL/LLOQ
2,3,7,8-TCDD	0.2	10	0.6	0.121	0.333	1	0.100	0.199	1			1	0.3		1	0.2	0.086	0.5
1,2,3,7,8-PeCDD	0.2	10	0.8	0.0708	1.67	5	0.129	0.258	5			5	1.5		5	1	0.232	2.5
1,2,3,4,7,8-HxCDD				0.107	1.67	5	0.207	0.413	5			5	1.5		5	1	0.547	2.5
1,2,3,6,7,8-HxCDD				0.108	1.67	5	0.234	0.468	5			5	1.5		5	1	0.497	2.5
1,2,3,7,8,9-HxCDD				0.119	1.67	5	0.222	0.443	5			5	1.5		5	1	0.723	2.5
1,2,3,4,6,7,8-HpCDD				0.144	1.67	5	0.270	0.54	5			5	1.5		5	1	0.327	2.5
OCDD				0.253	3.33	10	1.010	2.02	10			10	3		10	5	1.185	5
2,3,7,8-TCDF	0.406	600		0.112	0.333	1	0.120	0.239	1			1	1.5		1	0.2	0.105	2.5
1,2,3,7,8-PeCDF	-			0.0636	1.67	5	0.110	0.219	5			5	1.5		5	1	0.415	2.5
2,3,4,7,8-PeCDF	0.3	200	200	0.0607	1.67	5	0.110	0.219	5			5	1.5		5	1	0.345	2.5
1,2,3,4,7,8-HxCDF	0.4	40		0.0637	1.67	5	0.252	0.504	5			5	1.5		5	1	0.281	2.5
1,2,3,6,7,8-HxCDF	-			0.0657	1.67	5	0.197	0.394	5			5	1.5		5	1	0.311	2.5
1,2,3,7,8,9-HxCDF				0.0692	1.67	5	0.277	0.554	5			5	1.5		5	1	0.5	2.5
2,3,4,6,7,8-HxCDF				0.0977	1.67	5	0.204	0.408	5			5	1.5		5	1	0.483	2.5
1,2,3,4,6,7,8-HpCDF				0.079	1.67	5	0.194	0.388	5			5	1.5		5	1	0.376	2.5
1,2,3,4,7,8,9-HpCDF				0.133	1.67	5	0.257	0.513	5			5	1.5		5	1	0.268	2.5
OCDF				0.251	3.33	10	0.715	1.43	10			10	3		10	5	0.95	5



-- = not available.

CUL = cleanup level—Table 17 of PH ROD.

EDL = estimated detection limit.

HpCDD = heptachlorodibenzo-p-dioxin.

HpCDF = heptachlorodibenzofuran.

HxCDD = hexachlorodibenzo-p-dioxin.

HxCDF = hexachlorodibenzofuran.

LLOQ = lower limit of quantitation.

MDL = method detection limit.

OCDD = octachlorodibenzo-p-dioxin.

OCDF = octachlorodibenzofuran.

pg/g = picograms per gram.

PeCDD = pentachlorodibenzo-p-dioxin.

PeCDF = pentachlorodibenzofuran.

PH ROD = Portland Harbor Record of Decision.

PQL = practical quantitation limit.

PTW = principal threat waste threshold—Table 21 of PH ROD.

RAL = remedial action level—Table 21 of PH ROD.

TCDD = tetrachlorodibenzo-p-dioxin.

TCDF = tetrachlorodibenzofuran.

#### REFERENCES:

[1] Table 17. Portland Harbor Superfund Site Record of Decision, Portland, Oregon. EPA Region 10. Seattle, Washington, January 2017; EPA. 2020. Errata #2 for Portland Harbor Superfund Site Record of Decision ROD Table 17. January 14.

<sup>[2]</sup>Table 21. Portland Harbor Superfund Site Record of Decision, Portland, Oregon. EPA Region 10. Seattle, Washington, January 2017.

0785.01.13, 12/16/2020, Tf\_LSR Page 3 of 3



Laboratory	Methods Available	Standard TAT	Price	Expedited TAT	EDD	Tier 2 Report	Tier 4 Report	Primary Accreditation
Vista Analytical Laboratory	1613B/8290A	3 weeks	\$700	1 week	Yes	Yes	Yes	ORELAP/DoD
Pace Analytical Services	1613B/8290/8290A	4 weeks	\$525	1 week	Yes	Yes	Yes	ORELAP
Eurofins/Test America	1613B/8290A	4 weeks	\$650	1 week	Yes	Yes	Yes	ORELAP/NELAP Florida
Bureau Veritas Laboratories	1613B/8290A	3 weeks	\$450	1 week	Yes	Yes	Yes	DoD/NELAP in various states
ALS Global	1613B/8290A	3 weeks	\$250	1 week	Yes	Yes	Yes	ORELAP/DoD/ISO 17025
SGS Axys	1613B/8290A	4 weeks	\$680	1 week	Yes	Yes	Yes	ISO 17025/CALA/Ecology/NELAP Florida
Pacific Rim Laboratories	1613B	3 weeks	\$500	1 week	Yes	Yes	Yes	Ecology/CALA
Cape Fear Analytical	1613B/8290	3 weeks	\$435	1 week	Yes	Yes	Yes	Various States/USDA/A2LA
Analytical Resources Inc.	1613B/8290A	3 weeks	\$590	1 week	Yes	Yes	Yes	ORELAP/Ecology/DoD
Ceres Analytical Laboratory Inc.	1613B/8290A	2 weeks	\$375	1 week	Yes	Yes	Yes	CA ELAP/ORELAP/Ecology
Alpha Analytical	No Response	No Response	No Response	No Response	No Response	No Response	No Response	No Response

CA ELAP = California Environmental Laboratory Accreditation Program.

CALA = Canadian Association of Laboratory Accreditation.

DoD = U.S. Department of Defense.

Ecology = Washington State Department of Ecology.

EDD = electronic data deliverable.

ISO = International Organization for Standardization.

NELAP = National Environmental Laboratory Accreditation Program.

ORELAP = Oregon Environmental Laboratory Accreditation Program.

TAT = turnaround time.

USDA = U.S. Department of Agriculture.



Laboratory	Contact	Experience	Date of Interview	Extract Cleanup	Portland Harbor Experience
Vista Analytical Laboratory	Jennifer Christmann, Martha Meir	Over 30 years with a focus on sediment and some of the most contaminated areas of the U.S.	10/23/2020	All method 1613B cleanups are available and used as necessary.	Yes.
Pace Analytical Services	Mary Christie	Over 30 years with many analytes; 20+ years of individual experience.	10/21/2020	All method 1613B cleanups are available and used as necessary.	Yes. Past and ongoing.
Eurofins/Test America	Chris Rigell, Melissa Davidson, Ryan Henry	50+ people, with more than half being there for 20 years.	10/22/2020	All method 1613B cleanups are available and used as necessary.	Not for dioxins/furans but for PCB congeners. Test America Sacramento laboratory did the dioxin/furan analyses.
Bureau Veritas Laboratories	Stephanie Pollen	36 years of experience.	10/26/2020	All method 1613B cleanups are available and used as necessary.	Yes.
ALS Global	Ron Martino, Corey Grandtis, Kristin Neir	Previously CAS lab (around since 2000 at least)—Dx/F lab was an acquisition. Over 10 years as ALS. Five people work in the specialty lab in Houston—lots of collaboration with ALS in Burlington.	10/21/2020	All method 1613B cleanups are available and used as necessary.	Yes.
SGS Axys	Nick Corso, Richard Grace	Thirty-six years with Nick Corso. PMs with over 30 years' experience.	10/23/2020	All method 1613B cleanups are available and used as necessary.	Yes.
Pacific Rim Laboratories	David Hope	Lab is 18 years old—formerly Axys/BV. David d/f since 1988. Fifteen-person lab. Specialized in high-res organics. His business partner developed d/f methods for CA gov.	10/21/2020	All method 1613B cleanups are available and used as necessary.	No.
Cape Fear Analytical	Chris Cornwell	CF has been around for ten years. Partnered with GEL group lab—largest DOE lab in country. They came from a lab that SGS purchased. Most folks have 20+ years. Chris has 40 in lab and 30 in d/f. Small lab—approximately 15 staff.	10/21/2020	All method 1613B cleanups are available and used as necessary.	Yes.
Analytical Resources Inc.	Sue Dunnihoo	Forty years of prep; 36 years' experience at ARI. Organics lab supervisor 36 years.	10/23/2020	All method 1613B cleanups are available and used as necessary.	Possibly ongoing, but the samples would be blinds. In the early 80s ARI did lots of work in PHSS.
Ceres Analytical Laboratory Inc.	James Hedin	No show.	10/22/2020	No show.	No show.
Alpha Analytical	Did not respond	Did not respond.	Did not respond	Did not respond	Did not respond.



Study	Year Sampled	Number of Samples Analyzed	Analytical Method (as reported)	Laboratories Used	How Were EMPCs Qualified?	River Miles	Dataset Reporting Limits	Comments
Remedial Investigation/Feasibility Study background reference area (river mile 15.3 to 28.4) <sup>(a)</sup>	1997-2010	770	1613B	Various	Unknown, possibly both	0.7 to 26.1	RDL	
2017 DEQ Upriver <sup>(b)</sup>	2017	9	EPA 1613B	ALS Global subbed dioxin/furan to ALS Houston, Texas Laboratory	EMPC = J	18.35 to 25.2	EDL and RDL	EMPCs qualified with "J." Some EMPCs also qualified "U," possibly because of batch method blank detections.
2018 Baseline Pre-RD Group <sup>(c)</sup>	2018-2019	1,210	EPA1613B	Test America, CA	EMPC = J	1.9 to 28.3	MDL and RDL/QL	
2018 Baseline EPA Split <sup>(d)</sup>	2018	17	HRSM01.2	Cape Fear Analytical	EMPC = J	8.5 to Upriver Reach	MDL and CRQL	All samples collected in PH except for two. One of those two came from the Downtown Reach and one from the Upriver Reach.
2018 DEQ Orphan <sup>(e)</sup>	2019	9	EPA 1613B	ALS Global, Burlington	EMPC = U	16.1 to 19.6	MDL	

-- = no information.

CRQL = contract-required quantitation limit.

DEQ = Oregon Department of Environmental Quality.

EDL = estimated detection limit.

EMPC = estimated maximum potential concentration.

EPA = U.S. Environmental Protection Agency.

HRSM = high-resolution Superfund method.

J = estimated.

MDL = method detection limit.

QL = quantitation limit.

RDL = reporting detection limit.

U = non-detect.

REFERENCES:

(a) Remedial Investigation/Feasibility Study (RI/FS) background reference area (river mile 15.3 to 28.4) sample data from the Final Portland Harbor RI/FS—Remedial Investigation Report (EPA, 2016a).

b)2017 DEQ Upriver sample data from the Final Field and Data Report—Upriver Reach Sediment Characterization (GSI & Hart Crowser, 2018).

<sup>&</sup>lt;sup>(c)</sup>2018 Baseline Pre-RD Group from the Pre-Remedial Design Footprint Report (AECOM & Geosyntec, 2019).

<sup>&</sup>lt;sup>(d)</sup>2018 Baseline EPA Split sampling database.

<sup>(</sup>e) 2018 DEQ Orphan data from the Field and Data Report—Upriver Reach Sediment Investigation (Hart Crowser, 2020).



	2016 Portland Harbor RI/FS	2017 DEQ Upriver	2018 Baseline Pre-RD Group
Validation Level:	All results in FS database are labeled with QA2Cat1 (1999 data) or QA2Cat2 (all remaining 1997, 1999, 2002-2010).	Not stated, no copy of DVM, but appears to be level II based on description	From QAPP: 10% Stage 4 validation, 90% EPA Stage 2A
Validator:	Integral Consulting, EcoChem (Round 2A, 3B), Laboratory Data Consultants (Round 1)	Hart Crowser	AECOM
Validation Guidance Referenced:	LWG Round 1: EPA. 2002. National Functional Guidelines for Chlorinated Dioxin/Furan Data Review. Final. U.S. Environmental Protection Agency, Office of Emergency and Remedial Response, Washington, DC. (August 2002).  LWG Round 2A: EPA. 1996. EPA Region 10 SOP for the Validation of Polychlorinated Dibenzodioxin (PCDD) and Polychlorinated Dibenzofuran (PCDF) Data. U.S. Environmental Protection Agency, Region 10, Environmental Services Division, Seattle, WA.  EPA. 1999. Laboratory Data Validation: Functional Guidelines for Evaluating Organic Analysis. U.S. Environmental Protection Agency, Hazardous Site Evaluation Division, Washington, DC.  EPA. 1999. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review. EPA 540/R-99/00801. U.S. Environmental Protection Agency, Office of Emergency and Remedial Response, Washington, DC. October, 1999.	EPA. 1995. EPA Region 10 SOP for the Validation of Method 1668 Toxic, Dioxin-like PCB Data. U.S. Environmental Protection Agency Region 10, Environmental Services Division, Seattle, WA. December 8.  EPA. 2002. Guidance on Environmental Data Verification and Data Validation. EPA QA/G-8. EPA/240/R-02/004. November.  EPA. 2011. USEPA National Functional Guidelines for Chlorinated Dibenzo-p-Dioxins (CDDs) and Chlorinated Dibenzofurans (CDFs) Data Review. EPA 540-R-11-016. September.	EPA Method 1613B: Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS (October 1994)  EPA Contract Laboratory Program National Functional Guidelines for High Resolution Superfund Methods Data Review (April 2016)



	2016 Portland Harbor RI/FS	2017 DEQ Upriver	2018 Baseline Pre-RD Group
General Validation Notes:	FS sediment database is from the RI SCRA database (collected up to 7/19/2010), updates posted via LWG through February 2011, NWN's EE/CA dataset provided in 2013, and Arkema EE/CA datasets provided in 2014.  (From RI/FS Appendix A) Per RI data selection rules, the FS database includes data with quality assurance approval code indicating a Category 1 level of data quality and either a level of validation of "QA1" or "QA2."  From 2016 Portland Harbor RI/FS page 2-66:  • Category 1. Category 1 data are of known quality and are considered acceptable for use in decision making for the Site. There is sufficient information on these data sets to confidently verify that the data, along with associated data qualifiers, accurately represent chemical concentrations present at the time of sampling.  • Category 2. Category 2 data are of generally unknown or suspect quality. The quality assurance and quality control (QA/QC) information shows that data quality is poor or suspect, or essential QA/QC data (e.g., surrogate recoveries, matrix spike/matrix spike duplicates) are either incomplete or lacking.  Based on 2014 RI/FS table 2.3-1, QA1 appears to be similar to Stage 2A and QA2 appears to be similar to Stage 4. However, round 2A document states that 10% of sediment data were "fully validated" and remaining were validated at Level 3 by EcoChem, Inc.	Validation Reports in Appendix E of the May 2018 Hart Crowser Final Field and Data report, but these were not provided to the Portland Harbor data portal.	Validation is based on the March 2018 Pre-RD QAPP. Confirmed that stage 4 and 2A DVRs are both present. Quality Assurance Project Plan, Portland Harbor Pre-Remedia Design Investigation and Baseline Sampling, Portland Harbor Superfund Site (March 2018), and the laboratory quality control (QC) limits
Dioxin/Furan Specific Notes:	The 1999 data designated as Category QA1 is from COE "Willamette April Sediment Quality Evaluation." All remaining dioxin/furan data are designated Category QA2.  The LWG Round 2A report states that data were validated with EPA National Functional Guidelines and region 10 SOPs. (EPA 1994, 1995, 1996, 1999).  FS database contains several datasets from 1997-2010. Dioxin/furan validation qualification throughout dataset might not be consistent.	The data file provided only a single field of results. Non-detect results (U) are EDLs or detection limits raised based on method blank detections. Some EMPCs were qualified by Hart Crowser as non-detect with "U" but these were associated with method blank detections. Remaining EMPCs were qualified with "J." Non-detect result in the final EDD are indistinguishable from original EDLs. (RDL is the final detection limit which is either the EDL or raised detection limit based on validation).	



	2018 Baseline EPA Split	2019 DEQ Orphan
Validation Level:	EPA Stage IV	Level II Validation
Validator:	EPA Region 10 Environmental Services Unit, OERA	Hart Crowser
Validation Guidance Referenced:	Quality Assurance Project Plan (QAPP) for Portland Harbor Oversight Split Samples, 06/04/2018  EPA Contract Laboratory Program Statement of Work for High Resolution Superfund Methods (Multi-Media, Multi-Concentration) HRSM01.2  EPA National Functional Guidelines for High Resolution Superfund Methods Data Review (EPA542-B-16-001)  Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use (EPA-540-R-08-005).	EPA 2002. Guidance on Environmental Data Verification and Data Validation, EPA QA/G-8. November 2002.  EPA 2011. USEPA National Functional Guidelines for Chlorinated Dibenzop-Dioxins (CDDs) and Chlorinated Dibenzofurans (CDFs) Data Review.  U.S. Environmental Protection Agency, Office of Superfund Remediation and Technology Innovation (OSRTI), Washington, DC. EPA 540-R-11-016. September 2011.  EPA 2017. USEPA National Functional Guidelines for Organic Superfund Methods Data Review. U.S. Environmental Protection Agency, Office of Superfund Remediation and Technology Innovation (OSRTI), Washington, DC. EPA-540-R-2017-002. January 2017.



	2018 Baseline EPA Split	2019 DEQ Orphan
General Validation Notes:	"RMDL - Portland Harbor 47975_PJHSL1 Validation and Review of HRMS Data_04-12-2019"  Missing three attachments: manual/electronic data review results, sample summary report, and data validation report - analytical sample listing	Validation reported in Appendix C of "6220-URSI-Field and Data Report_01-31-20"
Dioxin/Furan Specific Notes:	Results with "ion ratios outside criteria" (EMPC) are not additionally qualified. EMPCs were flagged by Cape Fear with * and were reported as detections by EPA. EDL/MDLs appear to be accurate.  RDLs represent the original MDLs, as EMPCs were not qualified as nondetect. MDLs represent the original MDLs.	EMPC-flagged 2378-TCDF,12378-PeCDF, 123478-HxCDF, 123678-HxCDF, and OCDF results in method blank were not evaluated against samples because they were considered "not detected" by the reviewer. This could introduce some positive bias for these results. MDL/EDL unaffected ALS Burlington reported EMPCs as "ion abundance ratio did not meet acceptance criteria" instead of EMPC, which is also an available flag.  Hart Crowser qualified all "R" flagged results (ion ratios did not meet positive identification criteria) with "UJ."  RDLs represent qualified results, including results qualified based on EMPCs. MDLs represent original laboratory MDLs.



> = greater than.

COE = U.S. Army Corp of Engineers.

DEQ = Oregon Department of Environmental Quality.

DVM = data validation memorandum.

DVR = data validation report.

EDD = electronic data deliverable.

EDL = estimated detection limit.

EE/CA = engineering evaluation/cost analysis.

EMPC = estimated maximum potential concentration.

EPA = U.S. Environmental Protection Agency.

FS = feasibility study.

HxCDF = hexachlorodibenzofuran.

LWG = Lower Willamette River Group.

OCDF = octachlorodibenzofuran.

OERA = Office of Environmental Review and Assessment.

PeCDD = pentachlorodibenzo-p-dioxin.

PeCDF = pentachlorodibenzofuran.

QA = quality assurance.

QA2Cat1 = quality assurance 2 category 1.

QA2Cat2 = quality assurance 2 category 2.

QAPP = quality assurance project plan.

QC = quality control.

RD = remedial design.

RDL = reporting detection limit.

RI = remedial investigation.

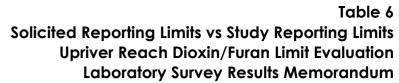
SCRA = site characterization and risk assessment.

SOP = standard operating procedure.

TCDF = tetrachlorodibenzofuran.



							,		Data Rev	iew Limits <sup>(3)</sup>			_		
Laboratory or Study:	PH	ROD Limi	ts		16 Portland Harbor /arious Laboratorie			2017 DEQ Upriver (ALS Houston)			Baseline Pre-RD G merica, West Sacra	•		118 Baseline EPA Sp ape Fear Analytic	
Reporting Limit (pg/g):	CUL <sup>(1)</sup>	RALs <sup>(2)</sup>	PTW <sup>(2)</sup>	GeoMean	50 Percentile	SEM	GeoMean	50 Percentile	SEM	GeoMean	50 Percentile	SEM	GeoMean	50 Percentile	SEM
2,3,7,8-TCDD	0.2	0.6	10	0.032	0.028	0.020	0.277	0.233	0.072	0.083	0.240	0.038	0.519	0.080	0.467
1,2,3,7,8-PeCDD	0.2	0.8	10	0.050	0.042	0.051	0.140	0.133	0.023	0.135	2.300	0.014	4.653	0.130	3.480
1,2,3,4,7,8-HxCDD				0.065	0.050	0.057	0.102	0.106	0.012	0.141	1.800	0.093	3.565	0.130	2.651
1,2,3,6,7,8-HxCDD				0.079	0.059	1.098	0.141	0.141	NA	0.125	1.500	0.142	3.036	0.120	2.255
1,2,3,7,8,9-HxCDD				0.064	0.056	0.069	0.224	0.229	0.026	0.117	2.900	0.142	5.853	0.110	4.361
1,2,3,4,6,7,8-HpCDD				0.755	0.815	0.205	NA	NA	NA	0.668	1.400	0.373	2.791	0.640	2.060
OCDD				4.618	4.600	1.300		NA	NA	0.540	3.400	0.171	6.835	0.500	5.084
2,3,7,8-TCDF	0.40658		600	0.091	0.120	0.012	0.172	0.137	0.047	0.131	0.360	0.023	0.831	0.130	0.675
1,2,3,7,8-PeCDF				0.044	0.033	61.660	0.135	0.125	0.029	0.160	2.000	0.067	4.687	0.150	14.490
2,3,4,7,8-PeCDF	0.3	200	200	0.035	0.029	0.060	0.185	0.182	0.045	0.171	1.100	0.075	2.493	0.160	7.539
1,2,3,4,7,8-HxCDF	0.4		40	0.064	0.045	5.116	0.200	0.244	0.054	0.261	1.900	0.117	3.848	0.240	2.891
1,2,3,6,7,8-HxCDF				0.052	0.035	28.730	0.165	0.144	0.058	0.261	2.300	0.110	4.653	0.230	3.480
1,2,3,7,8,9-HxCDF				0.042	0.037	0.012	0.124	0.129	0.020	0.228	1.800	0.087	3.991	0.200	8.753
2,3,4,6,7,8-HxCDF				0.042	0.034	0.036	0.146	0.155	0.036	0.190	1.200	0.091	2.351	0.170	1.752
1,2,3,4,6,7,8-HpCDF				0.163	0.190	0.118	0.930	1.040	0.308	0.365	2.700	0.100	5.498	0.335	4.100
1,2,3,4,7,8,9-HpCDF				0.069	0.056	0.045	0.118	0.122	0.011	0.433	2.700	0.102	5.483	0.400	4.100
OCDF				0.596	0.622	3.469		NA	NA	0.136	2.200	0.021	4.550	0.120	3.374





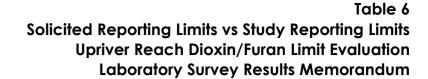
					Data Review Limits													
Laboratory or Study:	PH	ROD Limi	ts		2019 DEQ Orphan (ALS Burlington)		Pacific Rim Laboratories				SGS Axys		Bureau	Veritas Lat	ooratories	Vista Analytical Laboratory		
Reporting Limit (pg/g):	CUL <sup>(1)</sup>	RALs <sup>(2)</sup>	PTW <sup>(2)</sup>	GeoMean	50 Percentile	SEM	EDL	MDL	PQL/LLOQ	EDL	MDL	PQL/LLOQ	EDL	MDL	PQL/LLOQ	EDL	MDL	PQL/LLOQ
2,3,7,8-TCDD	0.2	0.6	10	0.086	0.070	0.025	0.043	0.01	0.2	0.05	0.25	0.8	0.111	0.181	1	0.20	0.269	0.5
1,2,3,7,8-PeCDD	0.2	0.8	10	0.043	0.039	0.014	0.013	0.05	1	0.05	0.53	2.5	0.105	0.242	5	0.23	0.729	2.5
1,2,3,4,7,8-HxCDD				0.061	0.044	0.023	0.008	0.09	1	0.05	0.69	2.5	0.09	0.211	5	0.30	0.654	2.5
1,2,3,6,7,8-HxCDD				0.055	0.061	0.011	0.006	0.1	1	0.05	0.49	2.5	0.097	0.228	5	0.39	0.593	2.5
1,2,3,7,8,9-HxCDD				0.054	0.043	0.013	0.007	0.06	1	0.05	0.6	2.5	0.094	0.203	5	0.39	0.619	2.5
1,2,3,4,6,7,8-HpCDD				0.063	0.057	0.032	0.013	0.09	1	0.05	0.86	2.5	0.098	0.177	5	0.43	0.615	2.5
OCDD				0.082	0.077	0.046	0.016	0.2	1	0.05	3.47	5.0	0.146	0.185	5	1.63	1.186	5
2,3,7,8-TCDF	0.40658		600	0.071	0.059	0.025	0.029	0.01	0.2	0.05	0.22	0.5	0.1	0.194	5	0.15	0.168	0.5
1,2,3,7,8-PeCDF				0.058	0.048	0.025	0.014	0.07	1	0.05	0.56	2.5	0.101	0.209	5	0.21	0.729	2.5
2,3,4,7,8-PeCDF	0.3	200	200	0.054	0.042	0.044	0.011	0.07	1	0.05	0.55	2.5	0.099	0.278	10	0.23	0.840	2.5
1,2,3,4,7,8-HxCDF	0.4		40	0.048	0.047	0.020	0.005	0.09	1	0.05	0.49	2.5	0.092	0.227	1	0.25	0.528	2.5
1,2,3,6,7,8-HxCDF				0.047	0.045	0.015	0.006	0.04	1	0.05	0.53	2.5	0.094	0.258	5	0.26	0.721	2.5
1,2,3,7,8,9-HxCDF				0.140	0.130	0.025	0.005	0.08	1	0.05	0.52	2.5	0.087	0.204	5	0.28	0.707	2.5
2,3,4,6,7,8-HxCDF				0.072	0.044	0.127	0.01	0.07	1	0.05	0.53	2.5	0.111	0.172	5	0.33	0.666	2.5
1,2,3,4,6,7,8-HpCDF				0.050	0.032	0.129	0.005	0.05	1	0.05	1.06	2.5	0.087	0.179	5	0.38	0.678	2.5
1,2,3,4,7,8,9-HpCDF				0.050	0.036	0.107	0.006	0.03	1	0.05	0.51	2.5	0.092	0.244	5	0.32	0.548	2.5
OCDF				0.058	0.043	0.023	0.009	0.17	2	0.05	1.18	5.0	0.108	0.743	10	0.62	4.507	5



Laboratory or Study:	PH	ROD Limi	ts	Analyi	Analytical Resources, Inc.			Cape Fear Analytical			Pace Analytical Services			fins/Test Ar	merica	ALS Global			
Reporting Limit (pg/g):	CUL <sup>(1)</sup>	RALs <sup>(2)</sup>	PTW <sup>(2)</sup>	EDL	MDL	PQL/LLOQ	EDL	MDL	PQL/LLOQ	EDL	MDL	PQL/LLOQ	EDL	MDL	PQL/LLOQ	EDL	MDL	PQL/LLOG	
2,3,7,8-TCDD	0.2	0.6	10		0.14	1	0.121	0.333	1		0.199	1			1	0.3		1	
1,2,3,7,8-PeCDD	0.2	0.8	10		0.18	1	0.0708	1.67	5		0.258	5			5	1.5		5	
1,2,3,4,7,8-HxCDD					0.18	1	0.107	1.67	5		0.413	5			5	1.5		5	
1,2,3,6,7,8-HxCDD					0.15	1	0.108	1.67	5		0.468	5			5	1.5		5	
1,2,3,7,8,9-HxCDD					0.22	1	0.119	1.67	5		0.443	5			5	1.5		5	
1,2,3,4,6,7,8-HpCDD					0.56	2.5	0.144	1.67	5		0.54	5			5	1.5		5	
OCDD					4.3	10	0.253	3.33	10		2.02	10			10	3		10	
2,3,7,8-TCDF	0.40658		600		0.063	1	0.112	0.333	1		0.239	1			1	1.5		1	
1,2,3,7,8-PeCDF					0.15	1	0.0636	1.67	5		0.219	5			5	1.5		5	
2,3,4,7,8-PeCDF	0.3	200	200		0.15	1	0.0607	1.67	5		0.219	5			5	1.5		5	
1,2,3,4,7,8-HxCDF	0.4		40		0.14	1	0.0637	1.67	5		0.504	5			5	1.5		5	
1,2,3,6,7,8-HxCDF					0.18	1	0.0657	1.67	5		0.394	5			5	1.5		5	
1,2,3,7,8,9-HxCDF					0.21	1	0.0692	1.67	5		0.554	5			5	1.5		5	
2,3,4,6,7,8-HxCDF					0.11	1	0.0977	1.67	5		0.408	5			5	1.5		5	
1,2,3,4,6,7,8-HpCDF					0.21	1	0.079	1.67	5		0.388	5			5	1.5		5	
1,2,3,4,7,8,9-HpCDF					0.16	1	0.133	1.67	5		0.513	5			5	1.5		5	
OCDF					1.1	2.5	0.251	3.33	10		1.43	10			10	3		10	



Laboratory or Study:	PH	ROD Limi	ts	Ceres A	Analytical Lo	aboratory	Alpha Analytical						
Reporting Limit (pg/g):	CUL <sup>(1)</sup>	RALs <sup>(2)</sup>	PTW <sup>(2)</sup>	EDL	MDL	PQL/LLOQ	EDL	MDL	PQL/LLOQ				
2,3,7,8-TCDD	0.2	0.6	10	0.2	0.086	0.5	NA	NA	NA				
1,2,3,7,8-PeCDD	0.2	0.8	10	1	0.232	2.5	NA	NA	NA				
1,2,3,4,7,8-HxCDD				1	0.547	2.5	NA	NA	NA				
1,2,3,6,7,8-HxCDD				1	0.497	2.5	NA	NA	NA				
1,2,3,7,8,9-HxCDD				1	0.723	2.5	NA	NA	NA				
1,2,3,4,6,7,8-HpCDD				1	0.327	2.5	NA	NA	NA				
OCDD				5	1.185	5	NA	NA	NA				
2,3,7,8-TCDF	0.40658		600	0.2	0.105	2.5	NA	NA	NA				
1,2,3,7,8-PeCDF				1	0.415	2.5	NA	NA	NA				
2,3,4,7,8-PeCDF	0.3	200	200	1	0.345	2.5	NA	NA	NA				
1,2,3,4,7,8-HxCDF	0.4		40	1	0.281	2.5	NA	NA	NA				
1,2,3,6,7,8-HxCDF				1	0.311	2.5	NA	NA	NA				
1,2,3,7,8,9-HxCDF				1	0.5	2.5	NA	NA	NA				
2,3,4,6,7,8-HxCDF				1	0.483	2.5	NA	NA	NA				
1,2,3,4,6,7,8-HpCDF				1	0.376	2.5	NA	NA	NA				
1,2,3,4,7,8,9-HpCDF				1	0.268	2.5	NA	NA	NA				
OCDF				5	0.95	5	NA	NA	NA				





-- = no data available.

CUL = cleanup level.

DEQ = Oregon Department of Environmental Quality.

EDL = estimated detection limit.

EPA = U.S. Environmental Protection Agency.

FS = feasibility study.

GeoMean = geometric mean.

HpCDD = heptachlorodibenzo-p-dioxin.

HpCDF = heptachlorodibenzofuran.

HxCDD = hexachlorodibenzo-p-dioxin.

HxCDF = hexachlorodibenzofuran. LLOQ = lower limit of quantitation.

MDL = method detection limit.

NA = not applicable.

OCDD = octachlorodibenzo-p-dioxin.

OCDF = octachlorodibenzofuran.

PeCDD = pentachlorodibenzo-p-dioxin.

PeCDF = pentachlorodibenzofuran.

pg/g = picograms per gram.

PH ROD = Portland Harbor Superfund Site Record of Decision.

PQL = practical quantitation limit.

PTW = principal threat waste threshold.

RAL = remedial action level.

RAO = remedial action objective.

SEM = standard error of the mean.

TCDD = tetrachlorodibenzo-p-dioxin.

TCDF = tetrachlorodibenzofuran.

REFERENCES:

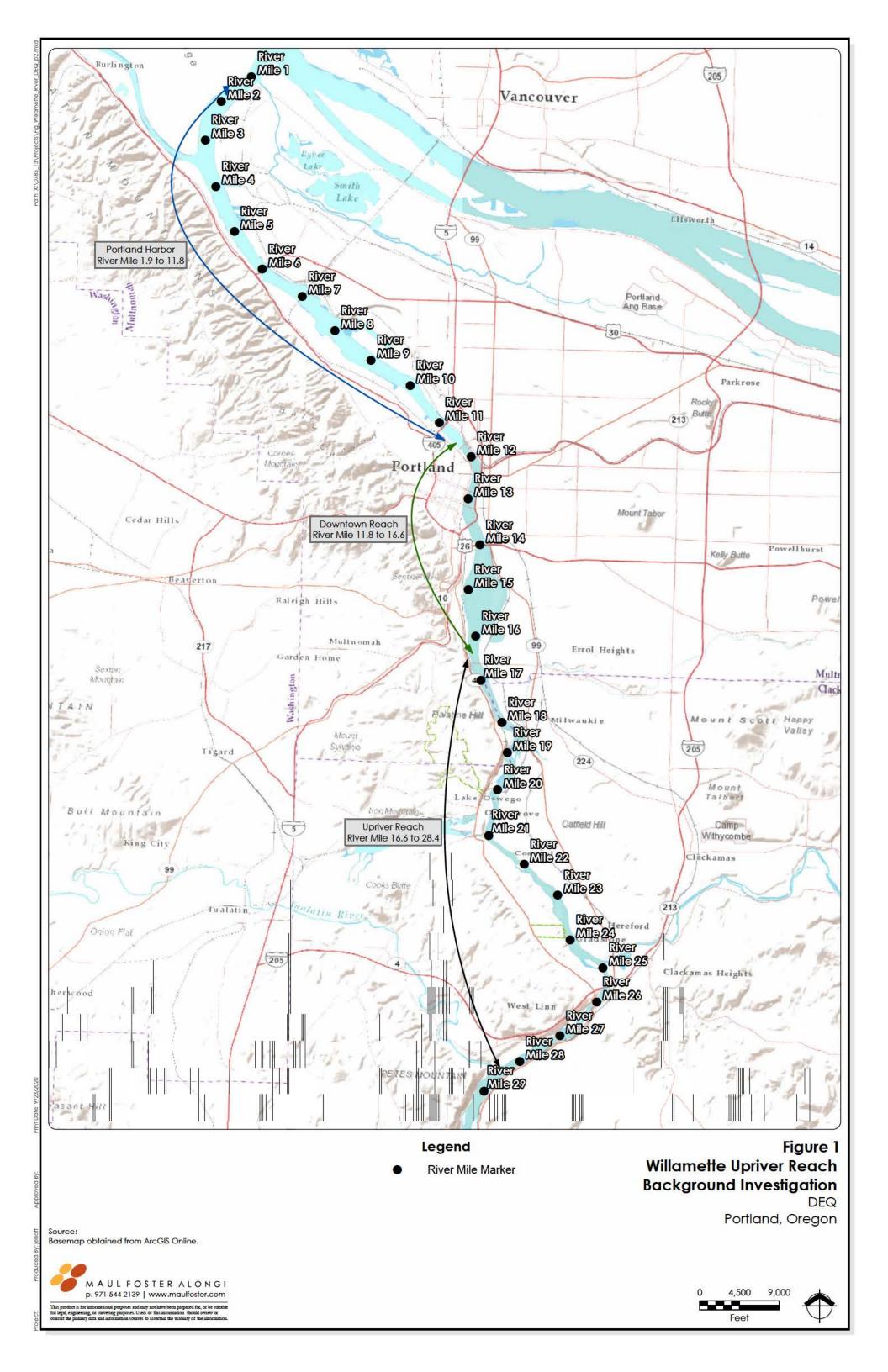
11/Table 17. Portland Harbor Superfund Site Record of Decision, Portland, Oregon. EPA Region 10. Seattle, Washington, January 2017; EPA. 2020. Errata #2 for Portland Harbor Superfund Site Record of Decision ROD Table 17. January 14.

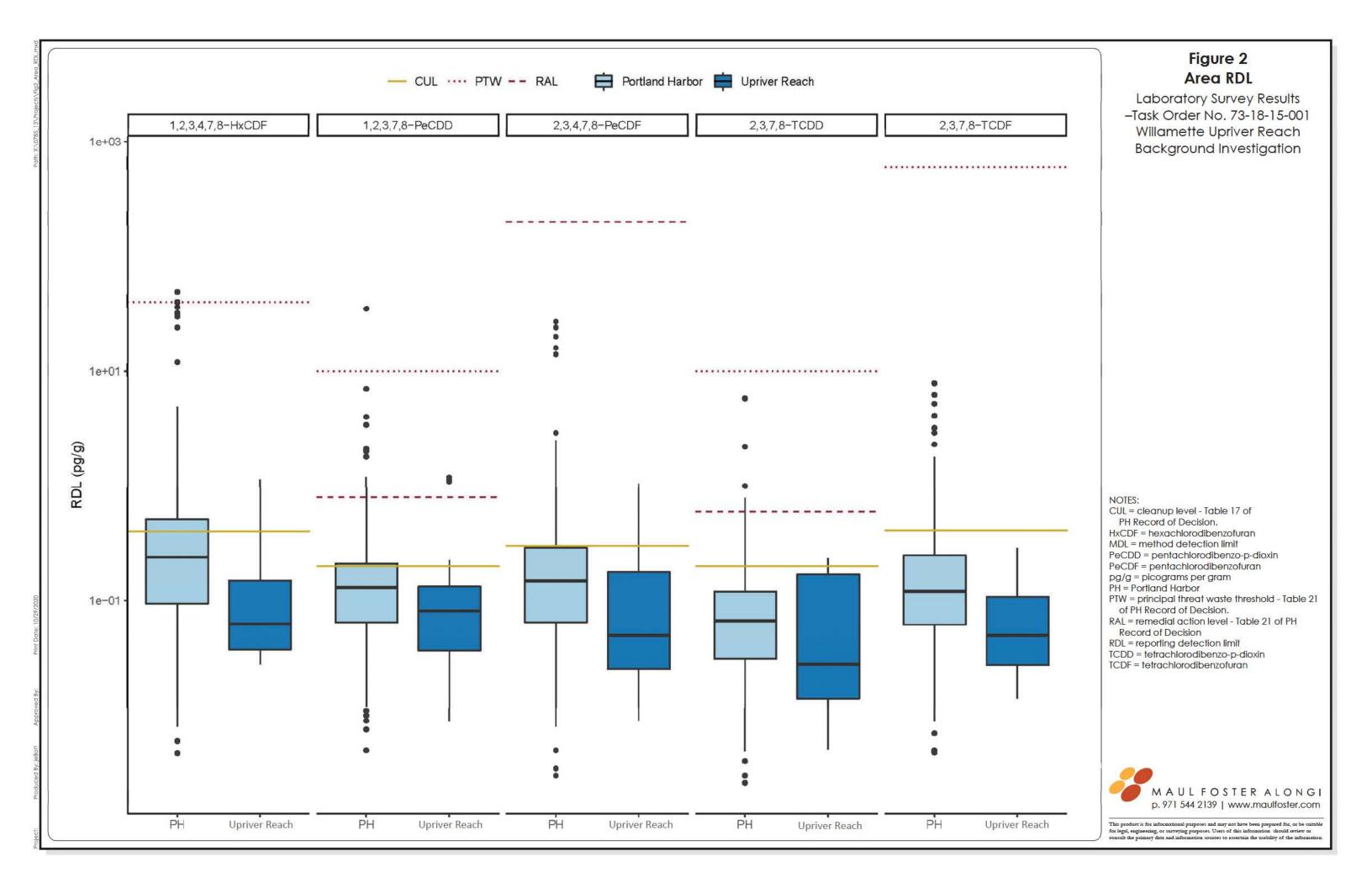
<sup>(2)</sup>Table 21. Portland Harbor Superfund Site Record of Decision, Portland, Oregon. EPA Region 10. Seattle, Washington, January 2017.

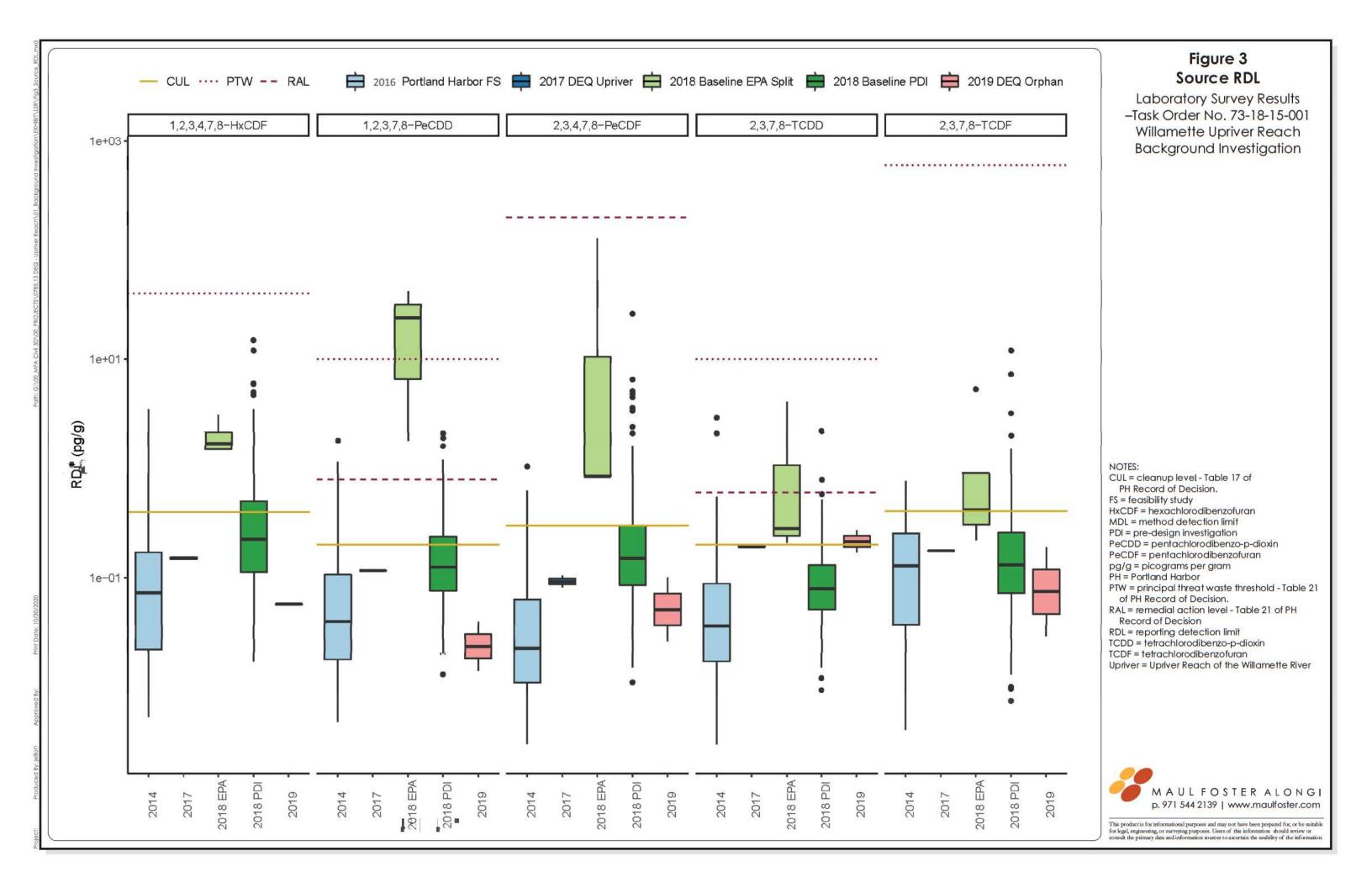
<sup>(3)</sup>See Dataset Reporting Limit column of Table 4 for specific limit type.

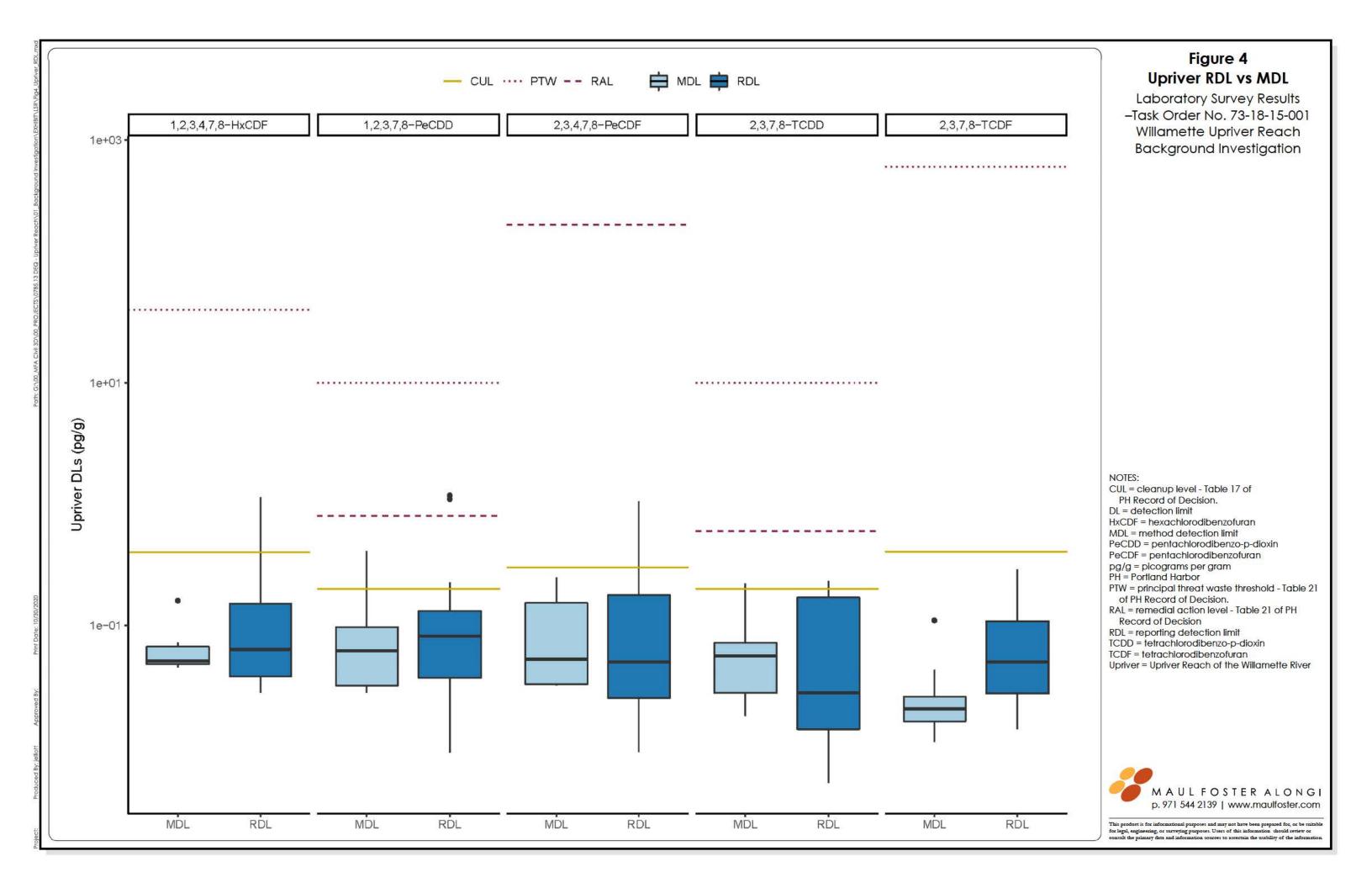
# **FIGURES**











# APPENDIX C EXAMPLE CHAIN-OF-CUSTODY FORMS



### APEX LABS

### **CHAIN OF CUSTODY**

Lab#	COC	of

6700 SW Sandburg St., Tigard, OR 97223 Ph: 503-718-2323

Company: Project Mgr:							Project Name:									Project #:											
Address:						Phone	Phone: Email:									PO #											
Sampled by:						ANALYSIS REQUEST																					
Site Location:														ţ					Mg, Mg, , TI,								
OR WA CA					S					ş		ist		ıll Lis				<u>8</u>	Cd, Cd, Hg, Hg, Na								Ì
AK ID	#			<u> </u>	# OF CONTAINERS	HCID	I-Dx	I-Gx	EX	8260 RBDM VOCs	8260 Halo VOCs	8260 VOCs Full List	8270 SIM PAHS	8270 Semi-Vols Full List	Bs	st	RCRA Metals (8)	Priority Metals (13)	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Hg, Mg, Mn, Mo, Ni, K, Se, Ag, Na, Tl, V, Zn TOTAL DISS TCLP	TCLP Metals (8)							ſ
SAMPLE ID	LAB ID#	DATE	TIME	MATRIX	# OF CO	NWTPH-HCID	NWTPH-Dx	NWTPH-Gx	8260 BTEX	8260 RE	8260 Ha	8260 VC	8270 SI	8270 Ser	8082 PCBs	8081 Pest	RCRA 1	Priority	Al, Sb, A Cr, Co, C Mn, Mo, N V, Zn TOTAL	TCLP N							Archive
																											Ī
Normal Tur	n Around	Time (T.	AT) = 10	Busines	ss Days	S	SPECIAL INSTRUCTIONS:																				
TAT Requested (circle)	1 Day		2 Day 5 DAY		3 Day Ot	her: _				_																	
SAMP	ES ARE	HELD FO																									
RELINQUISHED BY: Signature:	Date:		RECEIV Signature		:	Date:						RELINQUISHED BY: Signature: Date:								RECEIVED BY: Signature: Date:							
Printed Name:	Time:		Printed N	Vame:				Time:				Printed Name: Time:							Printed Name: Time:								
Company:			Company	y:									Company:							Company:							



6740 Campobello Road, Mississauga, Ontario L5N 2L8

Phone: 905-817-5700 Fax: 905-817-5779 Toll Free: 800-563-6266

#### CAM FCD-01191/5 CHAIN OF CUSTODY RECORD of Invoice Information Report Information (if differs from invoice) Project Information (where applicable) Turnaround Time (TAT) Required Regular TAT (5-7 days) Most analyses Quotation #: Company Name: Company Name: PLEASE PROVIDE ADVANCE NOTICE FOR RUSH PROJECTS P.O. #/ AFE#: Contact Name: Contact Name: Rush TAT (Surcharges will be applied) Address: Site Location: Phone: Site #: Phone: Date Required: Email: Email: Site Location Province:\_\_ MOE REGULATED DRINKING WATER OR WATER INTENDED FOR HUMAN CONSUMPTION MUST BE SUBMITTED ON THE BUREAU VERITAS LABORATORIES' DRINKING WATER CHAIN OF Rush Confirmation #: CUSTODY Regulation 153 Other Regulations **Analysis Requested** Res/Park Med/ Fine Table 1 CCME Sanitary Sewer Bylaw Table 2 MISA Storm Sewer Bylaw Ind/Comm Coarse Table 3 Agri/ Other PWQO Table \_ Other (Specify) FOR RSC (PLEASE CIRCLE) Y / N REG 558 (MIN. 3 DAY TAT REQUIRED) nclude Criteria on Certificate of Analysis: Y / N CONTAINERS SAMPLES MUST BE KEPT COOL ( < 10 °C ) FROM TIME OF SAMPLING UNTIL DELIVERY TO BUREAU VERITAS DONOT DATE SAMPLED TIME SAMPLED SAMPLE IDENTIFICATION MATRIX (YYYY/MM/DD) (HH:MM) COMMENTS 9 1 2 3 4 5 9 10 **BV JOB #** RELINQUISHED BY: (Signature/Print) DATE: (YYYY/MM/DD) TIME: (HH:MM) RECEIVED BY: (Signature/Print) DATE: (YYYY/MM/DD) TIME: (HH MM)

			Billing Infor			de de	A	nalysis /	s / Container / Preservative						Chain of Custody	Pageof			
						Pres Chk											Pace A	nalytical® er for Testing & Innovation	
																	National Cent	er for Testing & Innovation	
Report to:	Email To:	up-												12065 Lebanon Rd Mount Juliet, TN 3712 Phone: 615-758-5858					
Project Description:		City/State Collected:													Phone: 800-767-5859 Fax: 615-758-5859				
Phone: Fax:	Client Project #	‡		Lab Project #		-										L#			
Collected by (print):	Site/Facility ID	#		P.O. #			-										Table # Acctnum:		
Collected by (signature):	221	ab MUST Be I		Quote #											Template:				
Immediately Packed on Ice N Y	Next Day	5 Day 10 Da	(Rad Only)	Date R	No.	•										TSR: PB:	ļ.		
Sample ID	Comp/Grab	Matrix *	Depth	Date	Time	Cntrs											Shipped Via:	Sample # (lab only)	
												<u> </u>		_					
				-															
			1		-	-						_							
SS - Soil AIR - Air F - Filter	Remarks:							1		pH		Tem	P		coc s	eal Pr	le Receipt Che esent/Intact:	ecklistNPYNYN	
GW - Groundwater B - Bioassay WW - WasteWater DW - Drinking Water	[4] (A. C.							Flow Other								COC Signed/Accurate: Bottles arrive intact: Correct bottles used:			
OT - Other																	If Applicabl		
Relinquished by : (Signature) Date:			Ti	Fime: Received by: (Signat						Trip Blai	nk Rece	ived: Y	es / No HCL / N TBR		Prese	rvatio	deadspace:YN on Correct/Checked:YN		
Relinquished by : (Signature)		Date:	Ti	ime:	Received by: (Signat	ure)				Temp: °C Bottles Receiv					If pres	ervation	on required by Login: Date/Time		
Relinquished by : (Signature)		Date:	T	ime:	Received for lab by:	eceived for lab by: (Signature)						Tim	ne:		Hold:	Condition: NCF / OK			